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NPDES Compliance Monitoring Inspector Training: Laboratory Analysis

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NPDES Compliance Monitoring Inspector Training

Laboratory Analysis

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This document is part of a set of five documents which make up the National Pollutant Discharge Elimination System (NPDES) Compliance Monitoring Training Program. The training modules were developed by the Environmental Protection Agency (EPA), Office of Water Enforcement and Permits (OWEP), to instruct NPDES inspectors in various aspects of conducting NPDES Compliance Monitoring Inspections. The five training modules in this set are: 1) Overview, 2) Legal Issues, 3) Biomonitoring, 4) Sampling Procedures, and 5) Laboratory Analysis.

The training module describes procedures for

The modules were designed to be used as a self-taught course or as the basis for a lecture course to supplement on-the-job training for NPDES Inspectors. The modules should be presented by experienced and knowledgeable staff who can answer any questions, discuss policies regarding the topic being presented, and conduct on-the-job training. Each module covers a specific aspect of a compliance inspection. Instructors for a particular module may be selected according to their expertise, and training sessions could be scheduled based on the needs, the resources, and the time available to the Region. The modules can be presented individually or as a complete package.

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NPDES COMPLIANCE MONITORING
INSPECTOR TRAINING MODULE
LABORATORY ANALYSIS

U.S. ENVIRONMENTAL PROTECTION AGENCY

ENFORCEMENT DIVISION
OFFICE OF WATER ENFORCEMENT AND PERMITS
COMPLIANCE BRANCH
JUNE 1981

DISCLAIMER

This module has been reviewed by the Office of Water Enforcement and Permits, U.S. Environmental Protection Agency, and approved for publication. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

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HANDOUTS

FOREWORD

The National Pollutant Discharge Elimination System (NPDES) Compliance Monitoring Inspector Training Modules were developed by the Environmental Protection Agency (EPA), Office of Water Enforcement and Permits (OWEP), to instruct NPDES inspectors in various aspects of conducting NPDES Compliance Monitoring Inspections.

The EPA Regions have identified a need for training materials to instruct new employees in conducting NPDES inspections. Training seminars that are currently offered either do not address the training needs of an NPDES inspector or are not available due to limited resources or conflicting course schedules. These training modules were developed to fill the Regions' need for in-house inspector training.

The objectives of the training modules are:

1. To acquaint new inspectors with the NPDES Compliance Inspection program;
2. To serve as a refresher course for experienced NPDES Inspectors;
3. To assure consistency in the NPDES Compliance Inspection program; and
4. To inform and instruct inspectors concerning new inspection procedures.

The modules were designed to be used as a self-taught course or as the basis for a lecture course to supplement on-the-job training. The modules should be presented by experienced and knowledgeable Regional staff who can answer any questions, discuss Regional policies regarding the topic being presented, and conduct on-the-job training.

The module format was chosen for this training program because of its flexibility. Each module covers a specific aspect of a compliance inspection. Instructors for a particular module may be selected according to their expertise, and training sessions could be scheduled based on the needs, the resources, and the time available to the Region. The modules can be presented individually or as a complete package.

An outline of information contained in the individual training modules is listed below. There are currently five NPDES Compliance Monitoring Inspector Training modules:

1. The Overview module gives the inspector an overview of the compliance program and a brief summary of the different types of compliance inspections.

FOREWORD (Continued)

2. The Legal Issues module outlines the legal issues which must be addressed during an inspection and legal information that will assist inspectors in performing their duties.

3. The Biomonitoring module outlines the principles of biomonitoring and the role of biological testing in the inspection program.

4. The Sampling module details the sampling procedures that an inspector uses when conducting a sampling inspection.

5. The Laboratory Procedures module outlines the procedures and information necessary for an inspector to perform an effective evaluation of a permittee laboratory.

The layout of the text of each module is on a half page so that students may include their notes with the text.

These training modules were developed for the Regions and are designed to be used by the Regions for in-house training. If these modules are to be a success, the Regions must participate in their ongoing development. This can be accomplished by providing EPA Headquarters with changes or information which Regional instructors or managers believe would improve the modules. The format of the modules can be updated and revised at OWEP as the need arises as they were developed and produced at EPA Headquarters. Cooperation and commitment to training by the Regions will promote the development of a useful training document.

These training modules were developed primarily for Regional NPDES Inspectors; but they are also available to other interested parties such as State offices, attorneys, other program offices, facility owners and operators, and members of the general public.

Comments, information, and suggestions to improve the modules should be addressed to the:

Technical Evaluation and Support Section (EN-338)
Office of Water Enforcement and Permits
U.S. Environmental Protection Agency
401 M Street, SW
Washington, D.C. 20460

Modules covering new topics may be added to the existing ones as the need arises. Subject suggestions for future modules should be sent to the above address.

Requests for training modules will be handled at the above address depending on available supplies.

I. INTRODUCTION

I. INTRODUCTION

An integral part of the National Pollutant Discharge Elimination System (NPDES) compliance inspection process is the evaluation of the permittee's laboratory to determine whether the permittee's laboratory procedures are followed and reported in a way that is proper and consistent with the NPDES permit requirements. This module has been organized to follow, step-by-step, the major components involved in thoroughly evaluating a laboratory. Specifically, the following sequence will be covered.

A. Preinspection planning

B. Conducting the initial meeting

C. Assessing the laboratory's sample control

D. Reviewing the laboratory's analytical methods

E. Comparing Discharge Monitoring Reports (DMRs) with laboratory records

F. Evaluating the laboratory's quality control techniques

G. Conducting an audit of the laboratory's facilities and equipment

H. Conducting the exit meeting and preparing the final report

Objective of Module

The objective of this module is to provide you with helpful hints for conducting a thorough evaluation of the permittee's laboratory sample control, analytical, and quality assurance procedures, as well as assessing the adequacy of the conditions of the laboratory facilities and equipment.

Handout 1:Glossary

II. PREINSPECTION PLANNING

Purpose

Note: See also the Legal Issues Training Module for notification and entry procedures.

Approach

Note: For inspection of contractor laboratories, arrangements should be made through the appropriate permittee to gain permission for the inspection and to establish the date and time. All other inspection procedures remain basically the same.

Additional sources of information are provided in the reference section as well as in the handouts. A glossary of terms that will be useful to you is provided in Handout 1.

II. PREINSPECTION PLANNING

A. Purpose

Proper preparation and planning prior to visiting a laboratory for evaluation are key components in the evaluation process. The purpose of the evaluation is to determine whether the laboratory is performing the analyses and reporting the results in a way that is proper and consistent with the NPDES permit requirements and applicable regulations. A good way to look at the evaluation process is to approach it as an audit, in which laboratory procedures are compared to correct or standard procedures and deviations are then noted, evaluated, and reported. This approach sets the stage for the type of preinspection activities you should perform.

B. Approach

First, it is necessary to know which procedures to evaluate. The first step is to divide the laboratory procedures into two categories: general and specific. General procedures are those that should be followed by all laboratories regardless of the terms of the discharge permit. Sample control procedures fall into this category. Specific procedures are those which apply only to the permit under evaluation. Before visiting the laboratory, you should familiarize yourself with both the general laboratory requirements, which are described later in this module, and the specific procedures that are applicable to the laboratory in question.

Sources of Information

C. Sources of Information

The main source of information about the company and its compliance with its permit is the permit or inspection file. BE SURE THAT THE COPY OF THE PERMIT THAT YOU ARE EXAMINING IS UP TO DATE.

Handout 2:Preliminary Questionnaire

Send the permittee a preliminary questionnaire well in advance of the planned inspection (see Handout 2). Review the completed questionnaire in conjunction with the current permit.

Note: Use all or part of the questionnaire as dictated by the particular permittee.

Reference: Methods for Chemical Analysis for Water and Wastes

The permit contains the pollutant parameters to be analyzed and the type and frequency of sampling. You should check the permit to determine the type of samples being taken. Once you have determined the parameters to be analyzed, check the EPA manual entitled Methods for Chemical Analysis of Water and Wastes, EPA (1979) and 40 CFR 136 for a description of the appropriate standard analytical methods as determinted from the preliminary questionnaire. It is also useful to examine DMR's for the past 6 months and any DMR QA data and have them available during the inspection, as they may suggest parameters that are being analyzed incorrectly. Specifically, you should:

1. Record what pollutants have been reported to spot potential interferences
2. Spot general trends over the 6-month period
3. Check the average level of pollutant that could give you a clue regarding the

adequacy of the analysis. For example, if the level is less than or greater than that of similar plants, there may be potential problems.

Handout 3: Method of Analysis Breakdown

You should then divide each method into its component work elements. Handout 3 provides an example of this breakdown. You can also develop a chart for typical laboratory procedures that should be followed. This preinspection planning will provide you with a basis for comparing the subject laboratory procedures with those that are required.

Equipment

D. Equipment

Before leaving for your inspection visit, you should be equipped with the following:

1. Pencils and pens
2. Calculator
3. Notebook
4. Measuring tape
5. EPA Credentials
6. Protective (safety) equipment

The notebook, which should be bound and have numbered pages for legal reasons, is an important item in your kit. It will contain all of the notes you use to ensure the integrity of the information you gather during your laboratory evaluation (in case it should be needed as evidence later). It may be advisable to place all inspection information for a given plant into a single notebook, so that records can remain separate and organized. Each page should be signed, dated, and witnessed.

III. INITIAL MEETING

Objectives

Attendees

Handout 4: Outline for Initial Meeting

III. Initial Meeting

A. Objective

The initial meeting with company officials, which usually lasts one hour, plays a key part in the actual inspection. This meeting brings the concerned parties together and sets the tone for the rest of the inspection. The outcome of the initial meeting can determine whether the rest of the inspection is cordial and cooperative or difficult, and whether the laboratory to be inspected will be candid or attempt to conceal facts. Therefore, it is of utmost importance that you plan the conduct of the initial meeting with the goal of controlling it to your advantage.

The objectives of the initial meeting are twofold:

1. To obtain the cooperation of the laboratory
2. To obtain initial data for evaluating the laboratory

B. Attendees

Handout 4 provides an outline of things you should consider in conducting the initial meeting. First, you should consider the attendees. Handout 4 presents an optimal mix of people to be present at the initial meeting. These people include the laboratory officers to answer questions on policy and the laboratory directors to answer questions on procedures. This mixture can ensure both maximum cooperation and data in a minimum amount of meeting time.

Because the relationship between EPA and industry is often an adversary relationship from the beginning, it is important to make every effort to generate trust on the part of the laboratory personnel. The best way to achieve this trust is to allay their fears of an unknown process with unknown results. This can be accomplished by candidly and succinctly presenting the purpose of your visit and the process by which it is to be conducted. This opening statement is the second item on Handout 4.

Opening Statement

Outline for Initial Meeting (Handout 4)

C. Opening Statement

As shown in Handout 4, the opening statement should present a brief description of the purpose of the visit, how that purpose is to be achieved, what results the laboratory is to expect from your visit, and approximately when they will receive this information. It is also important to allow time at the end of the opening statement for laboratory personnel to ask questions about what is to happen and why. Be as forthright and candid in your answers as possible. In this way, the laboratory personnel can satisfy themselves that they have learned all they need to know; and consequently, their trust in EPA will be increased.

Questions to Ask

At this initial meeting you will need to ask questions to obtain the data to complete your report and forms and to obtain an impression of the general degree of laboratory personnel competence. Two types of questions to ask are:

1. General questions to assess how the laboratory handles the workflow

2. Permit-specific questions to assess how the laboratory

performs the work directly related to the plant permit.

General Questions

D. General Questions

General questions are intended to reveal overall laboratory policies and procedures that influence the overall quality of work performed. This quality affects all analytical data including those furnished in the plant's DMRs. One reason for doing this type of inspection is to help upgrade the quality of the data reported in DMRs. A list of general questions to be asked is:

1. Is there a Laboratory Methods Manual?

2. Is there a Laboratory Quality Control Manual?

3. Is there a Director of Quality Assurance?

4. Questions from EPA form 3560-3.
   a. Are EPA-approved analytical methods used?
   b. If alternate methods are used, has Regional approval been obtained for their use?
   c. What procedures are used to calibrate:
      1) Atomic Adsorption Spectrophotometer,
      2) Gas Chromatograph,
      3) pH Meters, and
      4) Specific Ion Detectors?
   d. Have the laboratory describe their quality control procedure.
   e. What percent of the laboratory sample load is duplicate samples?
   f. What percent of the laboratory load is spiked?
   g. Is the laboratory certified by the State or by any other certifying body?

5. Are acceptable standard reference materials used as a routine part of calibration and quality control programs?

6. What happens to a sample after it enters the laboratory?

Many of the questions asked at the initial meeting will be a review of the information contained in the preliminary questionnaire. This will provide direct verification of the answers provided to the questionnaire.

The first three general questions are intended to reveal whether the laboratory has written procedures for analysis and quality control, and whether there is a person who has responsibility for enforcing these procedures. Procedures are meaningless if they are not written, distributed, and practiced.

The fourth general question is a series of questions, which appear on the Compliance Inspection Report form (EPA 3560-3 page 3 of 4). These questions are intended to ascertain whether EPA-approved methods are being used for chemical analysis, whether adequate quality control (and calculation) procedures are in use, and whether the laboratory is certified by the State (or another body). These data are required by the inspection report form, and further serve to reveal the quality control methods in use by the inspection laboratory.

The fifth general question will quickly furnish the inspector with an indication of how seriously the laboratory regards quality in its standard and calibration reagents. Analysis of a known standard is required to standardize analytical methods. The known samples must be composed of materials of unimpeachable purity and product quality. An inspector should examine the standards used in calibration to assure that they are of appropriate quality. Appropriate quality refers to a standard that is either traceable to, or in fact is, a standard reference material (SRM) provided by National Bureau of Standards (NBS). Laboratories that use any other reagents as calibration standards are calibrating their methods against a standard of poorer quality and variable base.

The last general question will give you a view of the laboratory's internal sample control procedure. The issue of sample control is explored later in this module. Nonetheless, the initial meeting is the most appropriate time to raise the question. The inspector should receive an explanation of the laboratory sample control procedure and should request a copy of the written procedure. Lack of such a program makes reconstructing a past analysis impossible and is indicative of potentially deficient procedures in other work areas.

Specific Questions

E. Permit-Specific Questions

One of the major objectives of the laboratory visit is to assess the performance of the laboratory on analyses required by the plant's NPDES discharge permit. The permit-specific questions are listed below and intended to furnish you with this information.

1. What methods are used to analyze pollutant parameters in the plant permit?

2. Are these EPA-approved methods?
   a. If not, has approval been obtained from the EPA Region for their use?
   b. If not, has the analytical method been validated by an interlaboratory collaborative test?

3. Name the people who perform the analyses for the chemical found in the plant permits. (These people are to be visited during the laboratory tour.)

The purpose of the first question is to obtain information on the specific analytical methods for permit pollutants. The answer given by laboratory personnel should be in the form of method names with specific references (e.g., "4AAP" for phenols, "Cyanide by Distillation" for total cyanide), parameters measured, and frequency. The method names should be recorded in your notebook to be compared with the list of standard analytical test procedures in the Federal Register (40 CFR 136). Differences or discrepancies should be noted and discussed with the permittee.

The second question requires laboratory personnel to express whether they know if their analytical methods for permit pollutants are EPA-approved. Indications of confusion on this issue shows a lack of familiarity with EPA requirements, which, in itself, is fairly serious and may signal deviations from EPA-approved procedures.

Should the laboratory personnel answer "yes" to the inspector's question, this answer will be compared with answers to earlier method questions and to results of the laboratory tour to be conducted later in the visit. Should the laboratory personnel answer that their analysis procedures are not EPA-approved, the inspector should ask whether EPA-Regional approval has been obtained for using the substitute method. In the absence of Regional approval, the inspector should ask whether any other interlaboratory collaborative test, e.g., American Organization of Analytical Chemists (AOAC) has been used to validate the substitute method. The inspector may also check with EPA, Environmental Monitoring and Support Laboratory, Environmental Research Center, Cincinnati, Ohio, to request information on alternate method approval.

Lack of Regional approval for any substitute method is serious, and could result in enforcement action. Approval by an inter-laboratory test, while not a substitute for Regional approval, shows concern on the part of the laboratory for adequacy of the substitute analytical method and may be used as a basis for a later request for Regional approval.

The last permit-specific question asks for the names of the personnel who perform the analyses required by the discharge permit. You should interview these people while conducting the laboratory tour and ask them questions about how they perform the permit-required chemical analyses. You should record their answers in your notebook and compare these answers with the method descriptions reported in the approved source of procedures used in the lab such as the EPA Manual, Methods for Chemical Analysis of Water and Wastes, Environmental Monitoring and Support Laboratory, Environmental Research Center, Cincinnati, Ohio (1979).

Discrepancies between the two descriptions should be noted in your report.

Laboratory Self-Appraisal

F. Laboratory Self-Appraisal

Handout 5: Checklist for Laboratory Self-Appraisal

In addition to asking these questions at the initial meeting, it may be worthwhile to complete a checklist as shown in Handout 5. This checklist can be completed by you and the assembled laboratory personnel in approximately 30 minutes. This self-appraisal checklist is currently being used by private laboratories in other government programs and is an invaluable aid in assessing a laboratory's overall procedures and performance.

The checklist has numerical scores covering answers to questions involving general laboratory procedures for analysis, quality assurance, and quality control. The scoring system provides the inspector with a concrete numerical laboratory rating, which will provide invaluable support for conclusions reached in the inspection report. It is important for the laboratory personnel and the inspector to complete the checklist together, especially if the list is to be used as evidence in later legal or adjudicatory proceedings.

Laboratory Tour

G. Laboratory Tour

At this point, the initial meeting is ended; and the tour of the laboratory begins. The tour and later examination of logs and records will serve to support or refute conclusions reached concerning the adequacy of laboratory analytical procedures. The following sections of this module describe elements of the tour in detail.

IV. SAMPLE CONTROL PROCEDURES

OBJECTIVES

IV. SAMPLE CONTROL PROCEDURES

A. Objective

As mentioned earlier, an important part of the tour is to evaluate general procedures that are being followed regardless of the terms of the permit. The laboratory's sample control procedures fall into this category. While this type of system as applied to samples in enforcement activities is important from a legal stand-point, a similar laboratory sample control system is also useful for maintenance of the quality of analytical laboratory results. The importance of such a system should be emphasized to any permittee who does not have one.

Procedure

B. Procedure

A sample control system in a laboratory is a system that maintains records of persons handling a sample received by the laboratory, the dates of handling and (preferably) a brief description or comment section for each step in the process. The purpose of this type of system is to enable persons to reconstruct this handling process at a later date, in other words, after the sample has been analyzed.

Handout 6: Example of a Sample Control System

An example of a typical analytical process is given in Handout 6. The sample and analysis process is given on the left side of the handout. A suggested (many variations exist) documentation system is given on the right side. As the handout shows, the sample changes hands six times. Each time the sample changes hands, it must be recorded in order to reconstruct what happened to the sample while it was being analyzed. This reconstruction ability is necessary for the following reasons:

1. To establish that the analysis was performed
2. To trace the source of errors or unusual readings should it become necessary
3. To establish for later examination that requirements of a given analysis procedure, e.g., timing, were met

When examining the sample control procedure, you should assure yourself that the process can, in fact, trace the handling of an individual sample from the moment of its arrival in the laboratory to its final disposition.

Handout 7: Sample Control Form

A sample form is shown in Handout 7. This form is extremely simple and records only delivery and release of the sample. It relies on other analysis forms to record intermediate steps. The form can serve as the basis for reconstruction of procedure and, as such, is a useful tool.

When visiting a laboratory for inspection, ask the laboratory director if he has a sample control procedure. If he answers "no," then record this information in your notebook and try to determine why such a system does not exist (e.g., one is under development, too few samples are run, etc.). If he answers "yes," ask him to show you the write-up of the procedure, including the blank forms. After examining the blank forms and the procedural write-up, ask to examine at least 1 month's completed forms for actual samples from company files. Compare these forms against the company sample receiving log for the month. This 1-hour to 90-minute exercise will give you an indication of how rigorously company personnel follow the procedures. Note discrepancies in your notebook.

The next important check on sample control is to ask the laboratory director to literally walk you through the process for a typical sample. Ask each person you meet throughout the process what he does with a typical sample (or samples, if different types of samples have different processes). Follow the typical sample from beginning to end, including data handling and reporting procedures. Note the answers of each person you talk to and record these answers in your notebook. Later comparison of these answers with the written custody procedure will further reveal how rigorously the written procedure is followed.

The information you collect regarding the adequacy of the laboratory's sample control procedure can be valuable input for assessing the adequacy of the analytical methods.

V. ANALYTICAL METHODS

V. ANALYTICAL METHODS

In the next part of your tour, you should be concerned with the specific analysis procedures that are pertinent to the permit under evaluation.

In general, the analytical results produced by a permittee's wastewater laboratory are dependent upon the sampling procedures it employs. The best analytical methods, scrupulously applied, will be useless unless samples are taken properly. As part of a NPDES inspection, you should ascertain that good, consistent sampling procedures are followed by the permittee.

See the Module on Sampling Procedures for a detailed description of sampling procedures.

Analytical Methodology

A. Analytical Methodology

Uniform methods between cooperating laboratories are important in order to remove the methodology as a variable in comparison or joint use of data between laboratories. A lack of standardization of methods raises doubts as to the validity of the results reported. If the same constituent is measured by different analytical procedures within a single laboratory, or in several laboratories, the question is raised as to which procedure is superior, and why the superior method is not always used.

Analytical methods used by a permittee's laboratory should be selected by consulting 304(h) or EPA for approval of alternate methods.

Handout 8 & 9:
Standard Analytical
Test Procedures

References for
Standard Analytical
Test Procedures

Standardized analytical test procedures that have been promulgated under 40 CFR Part 136 as of May 1979 are listed in Handouts 8 and 9. These methods have been taken primarily from the following sources:

1. Methods for Chemical Analysis of Water and Waste (1979), Methods Development and Quality Assurance Research Laboratory, National Environmental Research Center, Cincinnati, Ohio 46268; U.S. Environmental Protection Agency, Office of Technology Transfer, Industrial Environmental Research Laboratory, Cincinnati, Ohio 45268.

2. Standard Methods for the Examination of Water and Waste Water, 14th Edition, (1976), American Public Health Association, 1015 18th Street, N.W., Washington, D.C. 20036

3. ASTM Annual Book of Standards, Part 31, Water (1975), American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.

4. "Methods for Collection and Analysis of Water Samples for Dissolved Minerals and Gases;" U.S. Geological Survey Techniques of Water-Resources Inventory, book 5, Ch. Al (1970). Brown, E., Skougstad, M.W., and Fishman, M.J.

It should be noted that publications of these sources are now being completed and the latest approved editions should be consulted.

As an EPA NPDES inspector, you should ascertain whether the permittee's laboratory is using the EPA-approved methods promulgated under 40 CFR Part 136 (Handouts 8 and 9). Handout 9 specifically provides the parameters, key elements of the method, and references of approved methods.

Approval of Alternate Methods

B. Approval For Alternate Methods

If any deviation from the use of approved methods is observed, the inspector should determine whether the permittee's laboratory has filed the proper application for alternate test procedures and received in writing the proper approval of alternate test procedures, as specified by 40 CFR.

Parts 136.4 and 136.5. Modifications of standardized analytical methods to provide specific improvements may be implemented only if the required written EPA approval has been obtained. You should ask to see the written approval in question. Handout 10 provides elements of a form that could be used by inspectors in documenting and checking the relevant analytical procedures.

Handout 10: Form for Documenting Analytical Procedures

Potential Problem Areas

C. Potential Problem Areas

As stated earlier, the major objective of this part of the tour is to verify whether the permittee's laboratory procedures are meeting the requirements for standard analytical procedures. As an aid in achieving this objective, Handout 11 provides a listing of potential problem areas associated with various analytical methods listed in the *Methods for Chemical Analysis of Water and Wastes*, EPA, (1979). You should include this handout in your notebook when checking the laboratory's analytical methods. Use of this Handout to evaluate permittee's analytical methods is helpful, due to the limited amount of time available for the inspection. If every procedure cannot be monitored, questions should be designed to evaluate the analyst's knowledge of difficult or complex steps.

Handout 11: List of Potential Problem Areas

Microbiological Procedures

The emphasis of this module is on chemistry since the majority of required permit parameters are physical/chemical parameters. In addition to these parameters, particular attention must be given to the microbiological procedures. For the unique requirements related to microbiology, refer to Chapter V

References: Mircobiological Procedures

(page 39) in the EPA Manual for the Interim Certification of Laboratories Involved in Analyzing Public Drinking Water Supplies, and Chapter 5, Part b (page 87) in the EPA Procedure for the Evaluation of Environmental Monitoring Laboratories. (See also Reference 9.)

VI. LABORATORY QUALITY ASSURANCE

VI. LABORATORY QUALITY ASSURANCE

Introduction

A. Introduction

See 40 CFR Section 122.7 (conditions applicable to all permits) which states that adequate laboratory and process controls including appropriate quality assurance procedures must be provided.

The effects of analytical methods on the precision/accuracy of reported data have been discussed in the previous section. However, there are many other sources of error that are independent of the analytical method. Examples of these are poor analyst technique, temporary voltage irregularities, bad reagents, etc. Unfortunately, the effects of many of these other sources cannot be predicted in advance. Very often a quality assurance program is the only way to correct these other sources of error or prevent them from affecting the overall precision/accuracy of the reported analytical results. Therefore, laboratory data should be supported with adequate documentation that provides valid records of the control measures applied to all factors bearing on final analytical results.

Definition

B. Definitions

Laboratory Quality Assurance

Laboratory quality assurance may be defined as a system of activities that provides confidence that overall laboratory quality control is effective. The system involves continual evaluation of the adequacy and effectiveness of the overall quality control program so that corrective measures can be initiated where necessary. For a specific analytical method, the program may involve verifications, audits, and the evaluation of the quality factors that affect the specifications, production, inspection, or use of the method.

Laboratory Quality Control

Laboratory quality control is the overall system of activities that provides a quality of analytical service to meet the user's needs. The aim of quality control is to provide data quality that is satisfactory, adequate, dependable, and economical. The overall system involves integrating the quality aspects of several related steps, including the proper specification of what is required, production to meet the full intent of the specification, inspection to determine whether the resulting product or service is in accordance with the specification, and review of usage to provide for revision of the specification.

Large sources of error are of great importance and must be controlled whereas time-consuming checks should be avoided when they account for only small sources of error.

Purpose of Quality Assurance

C. Purpose

A quality assurance program has two primary functions in the NPDES permittee's analytical laboratory. First, the program should continually monitor the reliability (accuracy, and precision) of the results reported, in order to answer the question, "How good are the results presented?" This function is the determination of quality. The second function is control of quality to meet the program requirements for reliability. For example, the processing of spiked sample provides a determination of measurement quality, while the use of analytical grade reagents is a control measure.

Just as every analytical method has a rigid protocol, quality control associated with a specific test must include definite required steps for monitoring the test and ensuring that its results are correct. For example, in a titration, standardization of the titrant on a frequent basis is an essential element of quality control. In any instrumental method of analysis, calibration and checking out of instrumental response are necessary quality control functions. As an EPA NPDES inspector, you should ascertain that experimental variables that affect the results of the analytical methods used by a permittee's laboratory are considered, evaluated, and controlled.

Policy and Objectives of Quality Assurance

D. Policy and Objectives

Each NPDES permittee's laboratory should have a written quality assurance manual that is distributed to all personnel responsible for analyses. The manual should clearly state the

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	objectives for producing data that meet user requirements in terms of specificity, completeness, precision, accuracy, representativeness, and comparability. Approximately 15 to 20 percent of each laboratory's resources should be devoted to its quality assurance program. The quality assurance manual should clearly identify the individuals involved in the quality assurance program and their responsibilities, and document the laboratory's standard operating procedures.
Elements of a Quality Assurance Plan	E. Elements of a Quality Assurance Plan
Procedures for Evaluating QA Plan	In evaluating the NPDES permittee's laboratory quality assurance plan, you should check to see that the following are included: 1. Provisions for the assurance of valid sampling 2. Use of EPA-approved standardized methodology in all cases 3. Control of services, instruments, equipment, and supplies 4. Maintenance of quality analytical performance 5. Efficient data handling and reporting procedures.
Key Element of QA Plan	The following key points should be addressed in the Quality Assurance Plan.
Sample Validity Note: See the Sampling Procedures Training Module for holding times.	Samples should be representative, properly collected, delivered in clean, appropriate containers, and preserved by approved measures. Analytical checks should be made on

containers and preservatives, and the appropriate sample holding times must be observed.

Sample Integrity

Written procedures for all aspects of sample handling should be distributed to all personnel concerned. For field samples, adequate seals, labels, and records should be kept for each container. Appropriate transport to the laboratory should be assured. The laboratory should have a sample coding specialist whose responsibility is to log in and code all samples.

Appropriate storage conditions and holding time must be observed for all samples. The laboratory should have formal systems for distributing analyses, for storage or discard after analysis, and for chain-of-custody documentation.

Documentation System

Complete and permanent records must be kept by all field and laboratory personnel. The required permanent records should be kept in bound log books with numbered pages whenever possible. Any procedures undertaken as quality checks, the results of any quality checks, and any checks by outside service personnel should be recorded, dated, and signed.

Quality Assurance Coordinator

Each laboratory should have a quality assurance coordinator who has overall responsibility for the development, implementation, and administration of the quality assurance program. The quality assurance coordinator should make a continuing assessment of the program, identify needs for accomplishing quality control training, and coordinate inter-laboratory quality control programs.

NOTES	LESSON
Control of Laboratory Services, Instruments, Equipment and Supplies	Elements of quality control that are affected by the use of laboratory services, equipment, supplies, reagents, etc. should be included in the overall plan. You should determine whether the laboratory is in conformance with the recommendations or requirements provided in this section. These elements should be checked carefully for any problems. This subject is pursued in more depth in the next section covering laboratory facilities and equipment.
Quality Assurance in Laboratory Facilities and Equipment	F. Quality Assurance in Laboratory Facilities and Equipment The inspector should question laboratory personnel to see that distilled water that is ammonia-free, carbon-dioxide-free, ion-free, and has a low organic background is available in the laboratory at all times. Dry, oil-free, uncontaminated compressed air should be available on a continuous basis. The laboratory should have efficient lighting and adequate constant voltage with appropriate grounding. Written requirements for daily warm up, standardization, calibration, and/or optimization procedures should be provided for all analytical instruments. Standards should be available to perform daily check procedures. Examples include standardized weights, certified thermometers,

filters or solutions for wavelength alignment checks, NBS or other standard reference materials with standard absorption curves, standard resistors, calibration solutions (buffers, conductivity standards, turbidity standards), analytical parameter standards to establish or to check calibration curves, and radioactive standards with date and count.

The laboratory should have written trouble-shooting procedures for instruments and written schedules for required replacement, cleaning, checks, and/or adjustments by service personnel.

The laboratory should use polyethylene bottles for solutions of boron, silica, and alkali, glass containers for organics, and brown glass containers for light-sensitive solutions.

Volumetric analyses should be performed only with Class A calibrated glassware. A standardized, consistent, mandatory program for cleaning glassware should be routine, with analytical checks on its effectiveness.

Required purity of laboratory reagents depends on the analyte, the sensitivity of the method, and the specificity of the detection system. If purity of reagents is not specified in the method, general inorganic analyses require analytical reagent (AR) grade chemicals (except that standardizing solutions must be made up with primary standard grade reagents), distilled water and solvents free of the analyte, and commercial grade gases. Flame emission and atomic absorbtion analyses of metal compounds require spectroquality reagents, distilled-in-glass acids, deionized distilled water, and commerical-grade, laboratory-supplied gases. Radiological analyses require scintillation grade reagents and solvents, and high-purity extra dry gases with low radioactive background. Organic analyses (e.g., gas chromatographic methods) require reference grade chemicals of highest purity. These reagents frequently necessitate cleanup procedures, e.g., cleaning using gas chromatographic methods.

Laboratory supplies with limited shelf life should be dated upon receipt. You should check to see that shelf life recommendations, including the discard date on the container and storage requirements, are being observed. The background reagents and solvents should be checked and method blanks should be run with every series of samples, or one for every nine samples. Purchasing high purity reagents with identical stock numbers will reduce the need to constantly check these background solutions and may prove cost effective. There should be written procedures for reagents and solvents describing limits of error, cleanup, hazard response methods, and application of correction procedures. Gas cylinders should be replaced at 100-200 psi.

Procedures for removing impurities in reagents should include recrystallization, precipitation, distillation, washing with solvents used in analysis, and aging for gases (radiological).

Reagents and standard solutions should be prepared with primary standard grade chemicals, careful weighing, class A volumetric glassware, appropriate quality distilled water or solvent; and a label listing compound(s) concentration, date of preparation or discard, and preparer should be attached. Dilute standards should be prepared at the time of use. Standard solutions should be restandardized as required by stability. Purchased solutions should contain the chemicals specified by the method and should be checked for accuracy. Clean containers of suitable composition with tight-fitting stoppers or caps should be used for storage. Safeguards should be taken against evaporation of solvents, absorption of gases and water vapor, and the effects of light or temperature. Reagents or solvents should be discarded when signs of discoloration, formation of precipitates, or significant changes in concentration are observed.

Quality Assurance in Sample Collection and Analysis

G. Quality Assurance in Sample Collection and Analysis

As stated in the NPDES compliance inspection guidelines, control checks should be performed by the inspector to ensure that sampling has been done properly. Many common analytical errors are caused by improper sampling, poor preservation, or lack of adequate mixing during compositing and testing. The following checks will help you and the laboratory coordinator determine when the sampling and analysis system is out of control.

Checks for Quality Assurance

Duplicate samples

Duplicate samples should be run to provide a proficiency check for precision.

NOTES	LESSON
Split Samples	Aliquots of the same sample may be run by both the permittee and EPA as a check on the permittee's laboratory procedures. Differences between EPA and permittee results can then be evaluated and the cause of the difference identified.
Performance Samples	Having the permittee analyze performance samples of permit parameters will also aid in identifying discrepancies in the permittee's analytical techniques and procedures. It is preferable to have these performance samples run prior to the visit so that problems can be discussed and corrected during the inspection.
Spiked Samples	Known amounts of a particular constituent should be added to an actual sample or blanks of deionized water at concentrations at which the accuracy of the test method is satisfactory. The amount added should be coordinated with the laboratory so that some samples are of an unknown value to those persons running the analysis. This will provide a proficiency check for analysis accuracy.
Sample Preservative Blanks	Acid and other chemical preservatives can become contaminated. The same quantity of preservative should be added to a sample of distilled water as would normally be added to the wastewater sample. This preservative blank is sent to the laboratory for analysis, and the blank is substracted from the sample value. Liquid chemical preservatives should be changed as often as necessary to prevent contamination.
Precision and Accuracy	H. Precision and Accuracy
	Precision refers to the reproducibility among replicate

observations. In a laboratory quality assurance program, precision is determined, not on the reference standards, but by the use of actual water samples which cover a range of concentrations and a variety of interfering materials usually encountered by the analyst. Obviously, such data should not be collected until the analyst is thoroughly familiar with the method and has obtained a reproducible standard curve. Accuracy refers to the degree of difference between observed and known, or actual, values. Again, accuracy should be determined with actual water samples routinely analyzed, and preferably, with the same series that is used in the precision determinations.

Handout 12: Precision and Accuracy

See Handout 12 for a graphical presentation of precision and accuracy.

To determine if the laboratory has established the precision and accuracy of their analytical procedures, you should verify that the following has been done annually (at a minimum), or when methods or the analyst have changed:

A minimum of seven replicates must be analyzed for each type of control check (duplicate samples, split samples, spiked samples, and sample preservative blanks). This information should be on record.

Quality Control Charts

Reference Chapter 6 of "Handbook for Analytical Quality Control in Water and Wastewater Laboratories" (EPA-600/4-79-019) for construction and use of control charts. Quality control charts were originally developed for the control of production processes where large numbers of items were being manufactured and inspected on an essentially continuous basis.

Description of Control Chart

A control chart consists of a graphical chart with the vertical scale plotted in units of the test result and the horizontal in units of time or sequence of results. The upper and lower control limits shown on the chart are used as criteria for action, or for judging the significance of variations between duplicate samples. The central line represents the average or the standard value of the statisical measure being plotted.

Daily precision and accuracy data can be plotted by means of quality control charts to determine whether valid, questionable, or invalid data are being generated from day to day. There are several techniques available for actually constructing quality control charts and plotting subsequent data. Two techniques currently used are the Shewhart technique and the CuSum technique (see "Handbook for Analytical Quality Control in Water and Wastewater Laboratories").

Precision Control Charts

Precision control charts are developed by collecting data from a minimum of 15 to 20 samples run in duplicate under assumed controlled conditions. These data should be generated over an extended period of laboratory time (e.g., 10 to 20 days).

Accuracy Control Charts

Accuracy control charts are developed by collecting data from a minimum of 15 to 20 samples, preferably spiked samples or standards under assumed controlled conditions. Again, these data should be generated over an extended period of laboratory time, and be representative of normal operating conditions. The determination of the control limits depends on the accuracy and precision of the procedure itself. It is common practice to set the limits at three standard deviations of the method.

Assuring Quality Analytical Performance

I. Quality Analytical Performance

Analyst

The key to quality analytical performance is the skilled analyst who has had appropriate and continuing training. An analyst should be willing to follow specified procedures, skilled in the manipulation of laboratory equipment and techniques required in analyses, and knowledgeable and skilled in performing the analyses for which he is responsible.

Establishing Analyst Precision

Seven replicates of four samples covering the concentration range of applicability should be run to establish analyst precision. Calculations for standard deviations for each set should be made. Results should be compared to the precision statement for the method and calculted in the same form. The precision statement can be found in the procedure for the method.

Establishing Analyst Accuracy

The following should be run to establish analyst accuracy except for gas chromatography and radiological instrumentation:

1. Spike a set of seven sample replicates having original concentrations low in the acceptable range of the method. The spike should bring the final concentrations to a level approximately twice the original concentration.
2. Spike a set of seven sample replicates having an original concentration mid-range in the acceptable range of the method. The spike should bring the final concentration to a level approximately 75%

of the upper limit of the method.

3. Precision and accuracy studies should be run among routine samples and cover 2 hours or more under normal operating conditions.

4. Split samples should be run and the results compared with those of another laboratory.

5. Unknown performance samples, available from EPA-EMSL, Cincinnati, at no charge, should be run periodically.

6. The laboratory should participate in round-robin method, performance evaluation studies, and laboratory evaluation programs.

Parameters Requiring Special Precautions to Assure Quality

Acidity/Alkalinity

J. Parameters Requiring Special Precautions

The laboratory must know how samples have been composited/preserved prior to delivery to the laboratory to assure sample validity. If the laboratory does the compositing /preservation, the following precautions must be considered. Compositing grab samples for acidity, alkalinity, and suspended solids analysis should not be done if a waste discharge varies outside the pH range specified in the NPDES permit. Mixing acid grab samples with neutral or basic grab samples changes the acidity/alkalinity relationship and results in a composite sample that may not be representative of the discharge during the compositing period. The acid-base reaction may also dissolve a portion of the inorganic solids.

Therefore, a discharge that varies outside the pH range specified in the NPDES permit should be analyzed for acidity, alkalinity, and suspended solids on an individual "grab" sample basis.

Dissolved Parameters

Samples for measuring dissolved parameters should be membrane filtered, using a 0.45μ filter, at the time of collection and composited under acidified conditions. Preservation should not be performed until after filtration.

Total Mercury

Samples for total mercury determination must be acidified at the time of collection. The addition of potassium dichromate will help stabilize dissolved mercury.

Phenolics and Cyanides

Simple phenolic compounds and free cyanide may be significantly degraded if not preserved at the time of sample collection. If the sample contains residual chlorine, it is also necessary to dechlorinate the sample prior to preservation, using ferrous sulfate as a dechlorination agent for phenolics and ascorbic acid for cyanide.

Sulfide and Sulfite

Sulfite samples must be analyzed in the field at the time of collection. Sulfides should be refrigerated at 4°C until analysis.

Performance Evaluations to Assure Quality

K. Performance Evaluation

At least two standards (high and low) should be analyzed daily with a blank at comparable operating conditions to verify an established standard curve.

Many methods, such as atomic absorption require daily preparation of a standard curve.

NOTES	LESSON
Reference: EPA Handbook for Analytical Water Quality Control	One of about every ten sample should be a duplicate to check precision according to acceptable standard deviation or % relative standard deviation (as given in th EPA Handbook for Analytical Water Quality Control (1979).
	One of about every 10 samples should be a spiked sample to check accuracy according to acceptable % recovery or % bias.
Documentation of Daily Performance	After about 20 sets of duplicate data results of spiked sample results have been collected, control charts for precision and accuracy, respectively, should be constructed to document the reliability of the data. The chart will show out-of-control numbers, trends toward out-of-control conditions, improved performance o an analyst, etc.
Secondary Checks on Performance	Quality control samples are available from EPA at no charge for many commonly analyzed constituents the concentrations are provided wit the samples. Such quality control samples should be run every 3-6 months at the time of sample collection whether the sample is a grab sample or a composite of grab samples.
Data Handling and Reporting to Assure Quality	L. Data Handling and Reporting
	As part of its quality assurance program, the NPDES permittee's laboratory must have a program for the systematic and uniform recording of data, and for processing and reporting it in proper form for interpretation and use.
Handout 13: Sample Bench Sheet	Handout 13 provides an example of a sample bench sheet.
Handling Analytical Values	Forms should contain correct calculation formulas reduced to simplest factors for quick, correct

calculations. Provisions should be made for cross-checking calculations. Rounding off rules should be uniformly applied, and significant figures should be established for each analysis and be present in the forms.

Data Processing

Note: All data should be readilly tracked from raw numbers to results given in the DMR

The laboratory should have developed report forms to provide complete data documentation and permanent records, and also to facilitate data processing. To avoid copying errors, the number of forms should be minimal. The inspector should check these procedures by following a few randomly selected values from laboratory records to recording on Discharge Monitoring Reports.

Reporting Data

The laboratory's program for data handling should provide data in the form/units required for reporting.

Data Storage

Data and laboratory records must be kept readily available to regulatory agencies for a specified length of time (three years for all NPDES data). A bound note-book or preprinted data forms permanently bound provide for good documentation.

VII. LABORATORY FACILITIES AND EQUIPMENT

VII. LABORATORY FACILITIES AND EQUIPMENT

An important factor in quality assurance is the maintenance of the laboratory's facilities and equipment. While they have been touched on briefly in the preceding section, the following section, which comprises the final part of your laboratory tour, will provide greater detail with regard to laboratory services, instruments, glassware, reagents, solvents, and gases.

Reviewing Laboratory Services

Handout 14: List of Laboratory Services

A. Laboratory Services

Handout 14 provides a list of laboratory services and the

NOTES	LESSON
	potential problems in supplying them. These services include distilled water, compressed air, and voltage-regulated electrical sources for delicate electronic instruments. Since many problems can be traced to poor services, you should note any problems or deficiencies found.
Examining Instruments	B. Instruments
	The modern analytical laboratory relies heavily upon instrumentation to generate reported data. Most modern analytical methods involve the use of one or more instruments. Therefore, a fundamental understanding of how the instruments work will help you in evaluating their use. While this information is available in any advanced analytical chemistry textbook, this section will present the major problems you may encounter when observing the analyst using the modern analytical equipment.
Handout 15: List of Instruments	Handout 15 lists the instruments most commonly used in routine water and wastewater analysis.
Analytical Balance	The analytical balance is the most important piece of equipment in any analytical laboratory. If the balance is not accurate, all gravimetric data and data related to weight-prepared standards will be in error.
	Most analytical balances currently used in well-equipped laboratories are "single-pan" balances. Since these instruments are fragile, subject to shock, temperature, and humidity changes, and of course, mishandling, you should note any indications of misuse. Calibration should be checked and recorded frequently,

using a 1-g or 0.1-g weight. There should also exist a label or separate record indicating annual sevice checks by outside representatives.

The high sensitivity of these balances requires that they be isolated from hot air ovens, mechanical shakers, flowhoods, and any other possible sources of interference.

pH Meter

A pH meter is used to measure the hydrogen ion concentration of a solution. A glass electrode is used as the indicator and a calomel electrode as the reference (single, combined electrodes are also available). Glass electrodes have a very fast response time in well-buffered solutions. However, in poorly buffered solutions, accurate readings are obtained slowly. This is particularly true when changing from buffered to unbuffered samples, as after standardization. The inspector should check to see that both glass and calomel electrodes are rinsed well with distilled water after each reading and rinsed or dipped several times into the next test sample before the final reading is taken. Weakly buffered samples should be stirred during measurement. Glass electrodes should not be allowed to become dry during periods of inactivity. When not in use, they should be immersed in distilled water.

The inspector should observe the analyst's performance in obtaining data from poorly buffered solutions. The analyst should allow enough time for the pH meter to come to equilibrium.

Conductivity Meters

These instruments measure the conductance of a solution. The conductance is expressed in mhos/cm. For natural water samples the reporting unit is micromhos/cm because of the small amount of current that can pass through. The sensing element in this conductivity meter is the conductivity cell.

During the inspection, cells should be examined to ensure that the plantinized coating of plates is intact, that plates are not coated with suspended matter or bent, distorted, or misaligned, and that lead wires are properly spaced. Measurements should be taken with known solutions to ensure accuracy.

Temperature has a pronounced effect on solution conductance, and must be corrected when results are reported. The specified temperature for reporting data used by most analytical groups and all EPA laboratories is 25°C.

Turbidimeters

Turbidimeters compare the intensity of light scattered by sample particles under defined conditions with the intensity of light scattered by a standard reference suspension. However, the lack of a primary standard for turbidity has resulted in an absence of uniformity among the available instruments. Correct use of the meter requires that samples containing turbidities in excess of 40 JTU be diluted to a value below this level and the results multiplied by the dilution factor. The inspector should check the condition of the sample cuvets to ensure that they are not scratched, since scratches would result in measurement errors. The cuvets should also be clean.

Spectrophotometers

In spectrophotometers, the sample solution absorbs electromagnetic radiation, and the amount absorbed is related to the concentration of the chemical in the solution. Absorption spectrophotometers can be divided into three types: ultraviolet (UV), visible, and infrared (IR).

The inspector should determine whether care is being taken to follow the manufacturer's instructions when using spectrophotometers. Colored solutions can be used for variable range instruments and organic compounds with known absorption curves can be used for UV and IR. A constant power supply is required to prevent flickering of the metal needle.

Care of absorption cells should be emphasized. All cells should be kept scrupulously clean and free of scratches, fingerprints, smudges, and evaporated film residues. Matched cells should be checked to ensure that they are equivalent by placing portions of the same solution in both cells and by taking several readings of the %T or OD values. If a cell is mismatched, it should be discarded or reserved for rough work.

Generally speaking, trained technicians may successfully operate any of the spectrophotometers. Because of the special techniques of sample presentation, instrument operation, and interpretation of absorption curves, the competence of the operator is important. Thus, the inspector should ascertain the qualifications of the individual who interprets UV readings.

Atomic Spectroscopic Instruments

Atomic spectroscopic instruments measure the absorption or emission of radiation by free atoms. Atomic absorption (AA) instruments are the most important in this group, and they are used for metal and semi-metal analysis.

There are a number of differences in the basic design of accessories for atomic absorption equipment. These include the light source, nebulizer burners, optical systems, readout devices, and mode conversion. For a program analyzing a wide variety of samples for a number of elements at varying concentrations, an instrument with maximum versatility is required.

Make sure standards are run at appropriate frequencies. In most cases, this means daily. The analyst should have a written check out procedure for the AA instrument and records showing when the check out was performed and what results were obtained.

- Many determinations suffer from potential interferences. This information should be readily available to the analyst so that the sample can be prepared correctly.

Organic Carbon Analyzer

In the organic carbon analyzer, material containing carbon is combusted to convert the carbon to carbon dioxide, which is then measured. Injection of a representative sample must be assured by the inspector.

Ion-selective Electrodes

The ion-selective electrode develops an electrical potential in response to the activity of the ion for which it is selective. For example, the probe designed to measure divalent cations does not respond to precipitated calcium and magnesium and, therefore, does not accurately measure total hardness.

Ion-selective electrodes are specific for the ion in question but can respond to other ions in high concentrations; therefore, care must be taken to eliminate interferences.

Gas-liquid Chromatoghraphs

The gas-liquid chromatograph is one of the most useful instruments available in the analytical laboratory. However, since special attention must be paid to the following items to obtain useful data from these instruments, observe or ask how the laboratory meets the following:

1. It is necessary to have accurate control of the column oven temperature. The temperature should be reproducible to 1°C, and have minimum gradients of 2°C throughout the oven. Most instruments on the market today meet these requirements if the instrument is properly maintained.

2. The stationary phase will bleed off the column at high temperatures resulting in a drifting baseline and possible changes in retention times. Keep the column below the maximum temperature of the stationary phase.

3. Before sample injection, the temperature of the column and injection port should be allowed to equilibrate.

4. Many organic compounds decompose when they come in contact with hot stainless steel. Therefore the injection block should be capable of accepting a quartz or glass tube to prevent the compounds from contacting hot metal.

5. Memory peaks can be eliminated or greatly reduced by using glass inserts for injection.

6. A precision, gas-tight, 10-microliter syringe that can be accurately filled, that will deliver reproducible injections, and that can easily and thoroughly be cleaned is recommended for gas chromatographic use. A Teflon plunger seal to prevent backwash and dead volume are desirable features. A gas-tight syringe is not required for liquid injections.

7. The septum should be changed at the end of each day's use. Changing at the end of each day allows overnight purging of the system of any bleed-off contaminants from the new septum. To avoid most of this bleed-off, the septa can be preconditioned by heating at 250°C in a vacuum oven for 2 hours.

8. The tritium detector has an upper limit of 225°C because of radioactive leakage. This temperature limitation makes the tritium detector susceptible to a buildup of high-boiling contaminants, (those that boil above 225°C) which reduce its sensitivity. The nickel detector can be operated up to 400°C to prevent this buildup. The detector should be kept at a higher temperature than the column to avoid contaminant buildup.

9. Nitrogen can be used for the flame ionization, electron

capture, flame photometric, and microcoulometric titration detectors. To avoid the risk of system or detector contamination from materials possibly present in the gas cylinder, the cylinder should be replaced when the tank pressure reaches 200 psi. It is also recommended that some type of gas purifier, composed of a molecular sieve, desiccant, and filter, be used on all combustion gases and carrier gases used with electron capture detectors.

Examining Glassware

C. Glassware

The measurement of trace constituents in water demands methods capable of maximum sensitivity. This requirement is especially true for metals and trace organics such as pesticides. Accuracy and precision in these methods cannot be attained unless special attention is given to glassware, particularly to prevent contamination. The purpose of this section is to acquaint the inspector with the kinds of glassware available, the correct use of volumetric ware, and various cleaning requirements. The inspector should request a description of glassware in use and cleaning operations and should observe these procedures being performed, where possible.

Types of Glassware

There are many grades and types of glassware available, ranging from student grade to others possessing specific properties such as resistance to thermal shock, alkali, low boron content, and super strength. Examples of some of these include:

1. Kimax- or Pyrex-brand glass, which is a relatively inert

all-purpose borosilicate glass

2. Vycor-brand glass, which is a silica glass (96%) made to withstand continuous temperatures up to 900°C and can be down-shocked in ice water without breaking
3. Corning-brand glass, which is claimed to be 50 times more resistant to alkalies than conventional ware and practically boron free (maximum 0.2%)
4. Ray-Sorb- or Low-Actinic-brand glass, which is used with light sensitive material
5. Corex-brand labware, which is harder than conventional borosilicates and better able to resist clouding and scratching

Volumetric Glassware

Volumetric glassware is accurately calibrated for precise measurements of volume. It includes volumetric flasks, volumetric pipets, and burets. Other less accurate types of volumetric glassware, such as graduated cylinders, are useful when exact volumes are not necessary. The volumetric apparatus must be read correctly; that is, the bottom of the meniscus should be tangent to the calibration mark. In order to reduce errors that might result from other sources such as temperature changes, Class A borosilicate glassware should be used.

Volumetric apparatus is calibrated "to contain" or "to deliver" a definite volume of liquid. This will be indicated on the apparatus with the letters "TC" (to contain) or "TD" (to deliver).

Volumetric flasks are calibrated to contain a given volume. They are available in various shapes and sizes ranging from a 1- to 2,000-milliliter capacity.

Volumetric pipets are calibrated to deliver or to contain a fixed volume of liquid. The usual capacities are 1 to 100 milliliters although micro-pipets are also available. Volumetric pipets should be held in a verticle positon when they are being emptied, and the outflow should be unrestricted. The tip of the pipet is placed in contact with the wall of the receiving vessel for a second or two after the free-flow has stopped. For "to deliver" pipets, it is important that the liquid remaining in the tip of the pipet is not blown out.

Burets are used to deliver definite volumes of liquid. The more common types that usually have a 25- or 50- milliliter capacity, are graduated to tenths of a milliliter, and are provided with stopcocks. (10-ml burets are commonly used for DO measurements.)

Federal Specifications for Volumetric Glassware

Circular 602 of the NBS, "Testing of Glass Volumetric Apparatus," describes the Federal specifications for volumetric glassware.

Manufacturers designate volumetric glass apparatus that meet the Federal specifications as Class A, and all such glassware is permanently marked (etched) with a large "A." In addition to the "A" marking and markings indicating the temperature at which the calibration was made, other markings also appear. These include the type of glass, such as Pyrex, Corex, Kimax, etc., the stock number of the particular item, and the capacity of the vessel. If the vessel contains a ground-glass connection, this will also be included along with the TD or TC symbol.

Fritted Ware

Porosity of fritted ware is controlled in manufacture, and the ware is individually tested and graded into six classifications according to pore size. This ware is less resistant to thermal shock than nonporous glassware.

Strong alkalies, strong hydrofluoric acid, and phosphoric acid, and scratching the surface may weaken or damage the fritted ware.

Cleaning Glassware

Reference: Handbook for Analytical Quality Control in Water and Wastewater Laboratories

Cleaning methods for glassware and porcelain should be appropriate for the substances that are to be removed and the determination to be performed, and should not damage the glassware or porcelain (see "Handbook for Analytical Quality Control in Water and Wastewater Laboratories").

Water-soluble substances are simply washed out with hot or cold water, and the vessel is finally rinsed with successive small amounts of distilled water. Other substances that are more difficult to remove may require the use of a detergent, organic solvent, dichromate cleaning solution, nitric acid, or aqua regia. To dry glass apparatus, the analyst should rinse with acetone and blow or draw air through it.

Absorption cells, used in spectrophotometers may be cleaned with detergent solutions to remove organic residues, but should not be soaked for prolonged periods in caustic solutions because of the possibility of etching. Organic solvents may be used to rinse cells in which organic materials have been used. Nitric acid rinses are permissible, but dichromate solutions are not recommended because of the absorptive properties of dichromate on glass. Rinsing and drying cells with alcohol or acetone before storage is a preferred practice. Cells used for analyses in the ppb range should be soaked in 1:1 solution of HCl and alcohol.

For certain determinations, especially trace metals, glassware should also be rinsed with a 1:1 nitric acid-water mixture. This operation should be followed by thoroughly rinsing with tap water and successive portions of distilled water. This may require as many as 12-15 rinses, especially if chromium is being determined. The nitric acid rinse is also important if lead is being determined.

Glassware used for phosphate determinations should not be washed with detergents containing phosphates. This glassware must be thoroughly rinsed with tap water and distilled water. For ammonia and Kjeldahl nitrogen, glassware must be rinsed with ammonia-free water.

Glassware used in determining trace organic constituents in water, such as chlorinated pesticides, should be as free of organic contaminants as possible. A chromic acid wash for at least 15 minutes is necessary to destroy these organic residues, followed by a thorough rinse with tap water and, finally, with distilled water.

VIII. EXIT MEETING AND FINAL REPORT

Introduction

Purpose of Exit Meeting

Use of the Deficiency Notice (EPA Form 3560-4)

Note: See the Overview Training Module for use of the Deficiency Notice

Final Report

VIII. EXIT MEETING AND FINAL REPORT

A. Introduction

Once you have completed your tour of the laboratory's facilities and equipment, you will have probably recorded the final input for completing your evaluation. You are now ready to consolidate your findings to arrive at an overall conclusion regarding the overall quality of data provided by the laboratory. Before leaving the facility, you should hold a closing conference or exit meeting with the laboratory officers and director.

B. Purpose

The purpose of the exit meeting is to provide the permittee with the following information:

1. A review of what you have done
2. A description of obvious problems that need to be corrected immediately
3. An explanation of steps you will take in reaching a supportable conclusion concerning the adequacy of the laboratory
4. A schedule for providing the results of your evaluation

C. Final Report

At your office, you will need to fill out the EPA Compliance Inspection Report form (EPA 3560-3) with a supplementary narrative, if necessary, documenting whether the subjects laboratory procedures were consistent and proper with the NPDES permit requirements. This should also be done promptly. You should also:

1. Compare the analytical methods observed with the standard approved procedures provided in Table I of 40 CFR 136, and handouts within this module
2. Compare the quality assurance procedures with those outlined in the Handbook for Analytical Quality Contol in Water and Wastewater Laboratories and in this module
3. Compare your observations on the state of the equipment with the information provided in this module
4. Note in your report any discrepancies observed in the above comparisons

IX. SUMMARY

IX. SUMMARY

This module has presented the components for conducting a thorough evaluation of a NPDES permitte's laboratory. A key ingredient of the evaluation is your advance preparation; namely, being familiar with chain-of-custody and quality control procedures and EPA approved analytical methods. By using the information presented in this module, you will be able to conclude whether the subject laboratory is, in fact, providing results that are consistent with the NPDES permit requirements.

HANDOUT 1

GLOSSARY

Accuracy—The degree of agreement between a measured value or a value calculated from measurements, and an accepted "true" value.

Buffer—A salt of a weak acid and a strong base, a salt of a strong acid and a weak base, or a combination of such salts that is capable of maintaining a desired pH value of a solution when substances of differing pH are added; the resistance to change of pH in buffers makes them useful as pH references.

Chain of Custody—A procedure that is established to record data on the handling of a sample from its generation to its final disposal.

Discharge Monitoring Report (DMR)—A report of effluent quality and quantity that is required to be filed by companies and municipalities that release such effluent to surface waters. This requirement arises from provisions of PL 92-500.

Infrared light—The range of wavelengths of the electromagnetic spectrum that is beyond the red end of the visible spectrum; from about 700 millimicrons to 25 microns.

Laboratory quality assurance—A procedure that is established to maintain a high standard of quality in a given process or function. (See pg. 20)

pH—A numerical expression of the acidity or alkalinity of a solution; it is the negative of the logarithm of the hydrogen ion concentration in moles per liter.

Precision—The degree of agreement between two or more measurements of the same quantity.

Reagent—A chemical of known purity used in laboratory work, of which quantities and identities of impurities are known.

Spectrophotometer—An instrument that measures the amount of light of chosen wavelengths or range of wavelengths that a sample absorbs.

Turbidity—The degree of opacity of a suspension due to the suspended particulates; proportional to the degree of light scattered by a suspension.

Ultraviolet light—The range of wavelengths of the electromagnetic spectrum that is beyond the violet end of the visible range; from about 450 to 200 millimicrons.

Visible light—The range of wavelengths of the electromagnetic spectrum that can be seen by humans; about 450 to 700 millimicrons (violet through red).

Analyte— The portion of the sample to be analyzed.

PRELIMINARY QUESTIONNAIRE

This questionnaire is designed to elicit all the information required prior to an onsite survey. Please make a concerted effort to furnish the information as accurately and concisely as possible.

For convenience, the questionnaire has been divided into six parts:

1) General Laboratory Information

2) Personnel

3) Laboratory Space and Facilities

4) Technical Services

5) Analytical Instruments and Special Apparatus

6) Quality Control

In each section, the questions are styled for the ease of the laboratory's response. In many cases only a check (✓) is required. Other questions call for a short answer; clarity and brevity should hallmark your response. If you need more space, please continue on blank sheets and attach them to the questionnaire.

Each section is independent, so that the different sections may be distributed to the most knowledgeable persons in the laboratory who can complete their parts independently. Finally, management can assemble and check all responses before returning the completed forms.

Upon return of the completed questionnaire, the onsite visit will be scheduled at your convenience. The time involved in the onsite evaluation can be minimized by a thorough presentation of the information sought in the preliminary questionnaire. Therefore, it is advantageous to both your laboratory and the evaluating agency if these questions are answered precisely and completely.

Thank you for your cooperation.

REGISTRATION FORM

The evaluation of Environmental Monitoring Laboratories is designed to assist the participating laboratories to upgrade their overall performance in order to safeguard the scientific and legal validity of their data. Submission of this registration form is the first step in the evaluation process. A preliminary questionnaire which requests background information about the laboratory's staff, facilities, and operating procedures is the second step. Upon completion of the preliminary questionnaire, an onsite visit to assess the performance capability of the laboratory will be scheduled at the convenience of the laboratory.

1. Name of Laboratory ____________________
2. Address ____________________
3. Telephone Number ____________________
4. Name of Laboratory Director ____________________
5. If Private, Name of Owner ____________________
6. Type of Laboratory ____________________
   - ☐ Commercial (privately owned, works on fee or contract basis)
   - ☐ Noncommercial (publicly controlled; usually does not work on a fee basis)
7. Provide a brief functional description of the activities of the laboratory ____________________
8. Media to be covered in evaluation
   - ☐ Water
     - ☐ Chemistry
     - ☐ Bacteriology
     - ☐ Biology
   - ☐ Air
   - ☐ Pesticides
   - ☐ Radiation
   - ☐ Other (specify) ____________________
9. If evaluation is not desired for complete analysis of any one of the media, list the specific tests for which you wish to be evaluated. An index of tests for which EPA approved methods are availiable is given on overleaf.* (Do not list for any medium for which you desire complete evaluation.)
10. Total Number of employees ________ Technical ________ Administrative ________

---

* EPA approved water and radiation test methods are referenced in Federal Register, Vol. 35, No. 199, October 16, 1973 Interim methods for algicides, chlorinated organic compounds, and pesticides can be obtained from Environmental Monitoring and Support Laboratory, U. S. Environmental Protaction Agency, 1014 Broadway, Cincinnati, Ohio 45268. EPA approved air test methods are referenced in Federal Register, Vol. 36, No. 228, November 25, 1971, and Vol. 38, No. 110, June 8, 1973.

Signature of Director ____________________ Date ____________

EML-01-9/75

PART 1. GENERAL INFORMATION ABOUT THE LABORATORY

1. Name of Laboratory ______________________________

2. Address ______________________________

3. Telephone Number ______________________________

4. Name of Laboratory Director ______________________________

5. Provide an organization chart of the laboratory, including any field operations or other internal affiliations to show how the laboratory fits into the general organizational structure. If attached, please check.

6. List names and addresses of external organizations used for significant supporting technical services.

______________________ ______________________

______________________ ______________________

______________________ ______________________

7. List names of principal users of services of the laboratory.

______________________ ______________________

______________________ ______________________

______________________ ______________________

8. Has the laboratory been evaluated previously? Yes ☐ No ☐ If yes, when __________ by whom __________

9. Do you perform monitoring activities? Yes ☐ No ☐ If yes, please check nature of monitoring activity:

☐ Water Quality
☐ Estuaries
☐ Oceans
☐ NPDES

☐ Air-Ambient
☐ Air-Source
☐ Pesticides

☐ Radiation
☐ Other (specify)
____

Lab Name ______________________

10. Do you participate in enforcement actions, emergency episodes, or special studies? Please specify.

____

____

11. Provide a copy of the latest annual report of the laboratory.

☐ Attached ☐ Not Available

Completed by ______________________ Date ______________

NAME TITLE

HANDOUT 2
~~(3 of 29)~~

Lab Name ______________________

PART 2. PERSONNEL

1. Laboratory staff. Complete Chart A for all technical personnel, including the laboratory director.

2. Provide brief summary job description for each supervisory, professional, and technical position. If attached, please check. ☐

3. What is the total number of laboratory employees? __________ Has this number increased over the past five years? Check if yes ☐

4. What portion of your staff participated in a formal training program related to improving work performance during the past year? Number __________ % __________

5. What was your turnover rate during the last 12 months?
   a) Administrative Staff Number __________ % __________
   b) Technical Staff Number __________ % __________

6. What portion of your staff was formally evaluated for performance during the past year?
   Number __________ % __________

7. What portion of your staff received merit increases in grade or salary during the past year?
   Number __________ % __________

8. What portion of your staff received service increases in grade or salary during the past year?
   Number __________ % __________

Completed by ______________________________ Date __________________
NAME TITLE

Lab Name ____________

CHART A

Complete Chart A for all technical personnel, including the laboratory director. Use a separate block for each employee and arrange the presentation to reflect the lines of organizational responsibility.

Date ____________ No. ____ of ____ pages.

Name	Training		Position	Years of Experience		Identify Analyses Performed by Numbers From Attached Index
	Degree (Circle One)	Major		Present Job	Previous Jobs	
	Ph.D. MS BS Assoc. HS					
	Ph. D. MS BS Assoc. HS					
	Ph. D. MS BS Assoc. HS					

Lab Name ____________________

PART 3. LABORATORY SPACE AND FACILITIES

CHART B

Complete Chart B. Please indicate both the availability and the adequacy of laboratory equipment and facilities.

Item	Description	Adequate		Additional Information
		Yes	No	
Buildings in Use Total m^2 (Sq. Ft.)				
Office Space Total m^2 (Sq. Ft.)				
Lab Space Total m^2 (Sq. Ft.)				
Bench-top Space Total m^2 (Sq. Ft.)				
Bench Hoods No. ____ Capacity (m/sec.) (lin. ft./min.)				

	Available		Adequate		Additional Information
	Yes	No	Yes	No	
Storage Space Chemicals					
Sample Storage - General					
Secured Space					
Refrigerated Space					
Hazardous Samples					
Controlled Space - Temperature					
Humidity					
Noise Insulation					
Shielded					
Clean Rooms					
Heat					
Air-Conditioning					
Electrical Services					
Gas					
Compressed Air					

Item	Available		Adequate		Additional Information
	Yes	No	Yes	No	
Vacuum					
Safety Equipment - Fire Alarm					
Fire Extinquishing Equipment					
Emergency Showers					
Eye Fountains					
Personal Equipment: glasses, gloves					
Hazardous Area Escape					
Flammable Material Storage					
Safety Cans					
Ventilation					
Smoking Areas					
Handling Equipment for Acids,					
Caustic					
OSHA Signs					
Water Supply - Distilled					
Deionized					
Ammonia - free					
CO_2 - free					
Bacteriologically Suitable					
Glassware Supply					
Glassware Washing Equipment					
Disposal Equipment - Broken Glass					
Contaminated Material, Solvents					
Library					
Conference Room					
Employee Lounge					
Employee Lockers					
Drinking Fountains					
Lunch Room					
Data Processing Equipment					

PART 4. TECHNICAL SERVICES OFFERED

Instructions

In Chart C, Table of Analytical Methods, you are asked to indicate the tests which are performed by this laboratory and the specific method(s) which you use for each test. This may be done simply by circling the appropriate references under Method Used in This Laboratory. In cases where you follow an EPA method which refers to ASTM or Standard Methods for the detailed procedure, you may circle the EPA reference only.

The Standard Methods, ASTM, and EPA references are given for your convenience. Standard Methods refers to Standard Methods for the Examination of Water and Wastewater, 13th Edition, 1971, published jointly by the American Public Health Association, the American Water Works Association, and the Water Pollution Control Federation. ASTM refers to the Annual Book of ASTM Standards, Part 31, Water, 1974, published by the American Society for Testing and Materials. EPA refers to Methods for Chemical Analysis of Water and Wastes, 1974, published by the Environmental Monitoring and Support Laboratory (National Environmental Research Center, Cincinnati, Ohio) and the Office of Technology Transfer, U.S. Environmental Protection Agency or to the Federal Register (for air tests). References in Standard Methods and ASTM are to method numbers, whereas references in the EPA Manual are to page numbers in the 1974 edition.

If this laboratory uses an alternate method or a modification of a referenced method, write "Other" under "Method Used in This Laboratory" and provide the requested information for each such case on a copy of **the form "Alternate Analytical Method", page** 40.

Under "Sample Frequency," please enter, in the #/Month column, the average number of samples per month tested by the specified method over the last 12 months. In the Peak Load column, give the maximum number of samples analyzed in a one-month period during the last 12 months. Your best estimates of these numbers will be satisfactory.

The tests listed in Chart C are limited to those referenced in the Federal Register. Referenced in the Federal Register but not included in Chart C are the variations in air methods suitable for measurement of emissions from stationary sources. Refer to Federal Register Vol. 36, No. 247, Part II, December 23, 1971; Vol. 38, No. 111, June 11, 1973; Vol. 39, No. 47, March 8, 1974; Vol. 40, No. 152, August 6, 1975; and Vol. 80, No. 194, October 6, 1975. There are, however, important areas not yet covered by such references. Biology is one such area. The bibliography appended to this procedure lists some of the sources of information on missing tests. Method 406, Standard Plate Count, is found in Standard Methods (Ref. 4). Refer also to the so-called "Equivalency Document," Federal Register, February 18, 1975.

	Available		Adequate		Additional Information
	Yes	No	Yes	No	
Logistic Services - Telephone					
Intercom					
Emergency Line					
Motor Vehicle					
Facilities as a Whole					

Completed by ______________________________ Date ____________________
NAME TITLE

Lab Name ____________________

1. Complete Chart C indicating analytical methodology which the laboratory wishes to have evaluated.
2. Provide a brief description of any special or unusual technical capability provided by the laboratory.

____

____

____

____

3. Provide a brief description of methods that you use for pretreatment of samples before analysis for trace metals, Tests No. 16-43

____

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Completed by ____________________ Date ____________________
NAME TITLE

At the end of Chart C a blank chart is included, page 20, on which information may be supplied on important tests performed by the laboratory which are not included in the check list.

CHART C. TABLE OF ANALYTICAL METHODS

Lab Name ______________

Test and Unit	Method	Method Used in This Lab			Copy Available	Sample Frequency	
		Circle Appropriate Reference[1] Check next col. if copies available in lab.					
		Standard Method	ASTM	EPA		#/ Month	Peak Load
General Analytical Tests:							
1. Alkalinity as $CaCO_3$ (mg $CaCO_3$/liter)	(a) Electrometric Titration, Manual	201	D1067-70B	p. 3			
	(b) Electrometric Titration, Automated	201		p. 3			
	(c) Automated, Methyl Orange			p. 5			
2. Biochemical Oxygen Demand (B.O.D.) 5-day 20° C (mg/liter)	(a) Modified Winkler with Full-Bottle	219		p. 11, 51			
	(b) Probe Method			p. 11, 56			
3. Chemical Oxygen Demand (C.O.D.) (mg/liter)	(a) Dichromate Reflux (organic C > 15 mg/liter)	220	D1252-67	p. 20			
	(b) Low Level Modification			p. 21			
	(c) Saline Water Modification (C1 > 2000 mg/liter)			p. 25			
4. Total Solids (Total Residue) (mg/liter)	(a) Gravimetric, Dried at 103-105° C	224A		p. 270			
5. Total Dissolved Solids (Total Filterable Residue) (mg/liter)	(a) Glass Fiber Filtration, Dried at 180° C	224E		p. 266			

Test and Unit	Method	Method Used in This Lab			Copy Available	Sample Frequency	
		Circle Appropriate Reference[1] Check next col. if copies available in lab.				# / Month	Peak Load
		Standard Method	ASTM	EPA			
6. Total Suspended Solids (Total Nonfilterable Residue) (mg/liter)	(a) Glass Fiber Filtration, Dried at 103-105° C	224C		p. 268			
7. Total Volatile Solids (Volatile Residue) (mg/liter)	(a) Gravimetric, Dried at 550° C	224B		p. 272			
8. Ammonia (as N) (mg/liter)	(a) Distillation and Titration			p. 159			
	(b) Distillation and Nesslerization			p. 159			
	(c) Distillation and Ammonia Electrode			p. 159, 165			
	(d) Automated Colorimetric Phenate Method			p. 168			
9. Total Kjeldahl Nitrogen (as N) (mg/liter)	(a) Digestion, Distillation & Titration	216		p. 175-181			
	(b) Digestion, Distillation & Nesslerization			p. 175-181			
	(c) Digestion, Distillation & Ammonia Electrode			p. 165, 175-181			
	(d) Automated Phenate Method			p. 182			
10. Nitrate (as N) (mg/liter)	(a) Cadmium Reduction Method (Nitrate-Nitrite)	213B	D992-71	p. 201			

Test and Unit	Method	Method Used in This Lab			Copy Available	Sample Frequency	
		Circle Appropriate Reference[1] Check next col. if copies available in lab.					
		Standard Method	ASTM	EPA		# / Month	Peak Load
10. Nitrate (as N) (mg/liter) (Cont.)	(b) Automated Cadmium Reduction Method (Nitrate-Nitrite)			p. 207			
	(c) Brucine Method			p. 197			
	(d) Automated Hydrazine Reduction Method			p. 185[2]			
11. Total Phosphorus (as P) (mg/liter)	(a) Single Reagent (Ascorbic Acid Reduction Method)	223CIII 223F		p. 249			
	(b) Automated Colorimetric Ascorbic Acid Reduction Method			p. 256			
	(c) Automated $SnCl_2$ Method	223E					
12. Acidity (mg $CaCO_3$/liter)	(a) Hydrogen Peroxide Digestion & Electrometric Titration		D1067-70E	p. 1			
	(b) Hydrogen Peroxide Digestion & Phenolphthalein End-Point Titration		D1067-70E				
13. Total Organic Carbon (T.O.C.) (mg/liter)	(a) Combustion and Infrared Method CO_2	138A	D2579-74	p. 236			
	(b) Combustion & Flame Ionization Method (CH_4)			p. 236			

Test and Unit	Method	Method Used in This Lab			Copy Available	Sample Frequency	
		Circle Appropriate Reference[1] Check next col. if copies available in lab.					
		Standard Method	ASTM	EPA		# / Month	Peak Load
14. Total Hardness (mg $CaCO_3$/liter)	(a) EDTA Titration	122	D1126-67B	p. 68			
	(b) Automated Colorimetric			p. 70			
	(c) Atomic Absorption (Ca + Mg)			p. 78-91, 103, 114			
15. Nitrite (as N) (mg/liter)	(a) Manual Colorimetric Diazotization			p. 215			
	(b) Automated Colorimetric Diazotization			p. 207*			

CHART C. TABLE OF ANALYTICAL METHODS

Lab Name: ______________

Test and Unit	Method	Method Used in This Lab Circle Appropriate Reference[1] Check next col. if copies available in lab. Standard Method	ASTM	EPA	Copy Avail-able	Sample Frequency # / Month	Peak Load
Tests for Trace Metals:							
16. Aluminum (mg/liter)	(a) Atomic Absorption	103A		p. 92 Ψ			
17. Antimony (mg/liter)	(a) Atomic Absorption			p. 94 Ψ			
18. Arsenic (mg/liter)	(a) Atomic Absorption (Gaseous Hydride Method)			p. 95 Ψ			
	(b) Gaseous Hydride - Silver Diethyldithiocarbamate Colorimetric	104A		p. 9			
19. Barium (mg/liter)	(a) Atomic Absorption	129A		p. 97 Ψ			
20. Beryllium (mg/liter)	(a) Atomic Absorption	129A		p. 99 Ψ			
	(b) Aluminon Method	106B					
21. Boron (mg/liter)	(a) Curcumin Method	107A		p. 13			
22. Cadmium (mg/liter)	(a) Atomic Absorption	129A	D2576-70	p. 101 Ψ			
	(b) Dithizone Colorimetric Method	211(II)B					
23. Calcium (mg/liter)	(a) Atomic Absorption		D2576-70	p. 103 Ψ			
	(b) EDTA Titration	110C		p. 19			

Test and Unit	Method	Method Used in This Lab Circle Appropriate Reference[1] Check next col. if copies available in lab. Standard Method	ASTM	EPA	Copy Avail-able	Sample Frequency # / Month	Peak Load
24. Chromium VI (mg/liter)	(a) Extraction and Atomic Absorption			p. 78-91, 105 Ψ			
	(b) Diphenylcarbazide Colorimetric	211(II)D					
25. Chromium, Total (mg/liter)	(a) Atomic Absorption	129A	D2576-70	p. 78-91, 105 Ψ			
	(b) Oxidation & Diphenylcarbazide Colorimetric	211(II)C	D1687-67	p. 105			
26. Cobalt (mg/liter)	(a) Atomic Absorption		D2576-70	p. 107 Ψ			
27. Copper (mg/liter)	(a) Atomic Absorption	129A	D2576-70	p. 108 Ψ			
	(b) Neocuproine Colorimetric	211(II)E	D1688-68				
28. Iron (mg/liter)	(a) Atomic Absorption	129A	D2576-70F	p. 110 Ψ			
	(b) O-Phenanthroline Colorimetric	211(II)F	D1068-68A				
29. Lead (mg/liter)	(a) Atomic Absorption	129A	D2576-70G	p. 112 Ψ			
	(b) Dithizone Colorimetric	211(II)G					
30. Magnesium (mg/liter)	(a) Atomic Absorption	129A	D2567-70	p. 114 Ψ			
	(b) Gravimetric	127A	D511-52				
31. Manganese (mg/liter)	(a) Atomic Absorption	129A	D2567-70	p. 116 Ψ			

Test and Unit	Method	Method Used in This Lab Circle Appropriate Reference[1] Check next col. if copies available in lab			Copy Available	Sample Frequency	
		Standard Method	ASTM	EPA		# / Month	Peak Load
32. Mercury (mg/liter)	(a) Flameless Atomic Absorption: Manual Cold Vapor Technique (Hg in Water)		D3223-73	p. 118 Ψ			
	(b) Flameless Atomic Absorption: Automated Cold Vapor Technique (Hg in Water) (not approved generally)			p. 127 Ψ			
	(c) Flameless Atomic Absorption: Manual Cold Vapor Technique (Hg in Sediment)		D3223-73	p. 134 Ψ			
33. Molybdenum (mg/liter)	(a) Atomic Absorption			p. 139 Ψ			
34. Nickel (mg/liter)	(a) Atomic Absorption		D2576-70	p. 141 Ψ			
	(b) Heptoxime Colorimetric	211(II)I					
35. Potassium (mg/liter)	(a) Atomic Absorption			p. 143 Ψ			
	(b) Colorimetric	147B					
	(c) Flame Photometric	147A	D1428-64				
36. Selenium (mg/liter)	(a) Atomic Absorption (Gaseous Hydride Method)			p. 145 Ψ			
37. Silver (mg/liter)	(a) Atomic Absorption	129A		p. 146 Ψ			

Test and Unit	Method	Method Used in This Lab			Copy Available	Sample Frequency	
		Circle Appropriate Reference[1] Check next col. if copies available in lab.					
		Standard Method	ASTM	EPA		# / Month	Peak Load
38. Sodium (mg/liter)	(a) Atomic Absorption			p. 147 Ψ			
	(b) Flame Photometric	153A	D1428-64				
39. Thallium (mg/liter)	(a) Atomic Absorption			p. 149 Ψ			
40. Tin (mg/liter)	(a) Atomic Absorption			p. 150 Ψ			
41. Titanium (mg/liter)	(a) Atomic Absorption			p. 151 Ψ			
42. Vanadium (mg/liter)	(a) Atomic Absorption			p. 153 Ψ			
	(b) Colorimetric (Catalysis of Gallic Acid Oxidation)	164A					
43. Zinc (mg/liter)	(a) Atomic Absorption	129A		p. 155 Ψ			
	(b) Dithizone Colorimetric Method	165B	D1691-67				

CHART C. TABLE OF ANALYTICAL METHODS

Lab Name: ____________________

Test and Unit	Method	Method Used in This Lab			Copy Available	Sample Frequency	
		Circle Appropriate Reference[1] Check next col. if copies available in lab.					
		Standard Method	ASTM	EPA		# / Month	Peak Load
Tests for Nutrients, Anions, and Organics							
44. Organic Nitrogen (as N) (mg/liter)	(a) Kjeldahl Nitrogen minus Ammonia Nitrogen	215		See (8) and (9) above			
45. Orthophosphate (as P) (mg/liter)	(a) Single Reagent Ascorbic Acid Reduction Method	223F	D515-72A	p. 249			
	(b) Automated Colorimetric Ascorbic Acid Reduction Method			p. 256			
46. Sulphate (as SO_4) (mg/liter)	(a) Gravimetric	156A	D516-68A	p. 283			
	(b) Turbidimetric	156C	D516-68B	p. 277			
	(c) Automated Colorimetric Barium Chloranilate			p. 279			
47. Sulfide (as S) (mg/liter)	(a) Titrimetric Iodine	228A		p. 284			
48. Sulfite (as SO_3) (mg/liter)	(a) Titrimetric Iodide-Iodate	158	D1339-72C	p. 285			
49. Bromide (mg/liter)	(a) Titrimetric Iodide-Iodate		D1246-68C	p. 14			
50. Chloride (mg/liter)	(a) Silver Nitrate	112A	D512-67B				
	(b) Mercuric Nitrate	112B	D512-67A	p. 29			
	(c) Automated Colorimetric Ferricyanide			p. 31			

Test and Unit	Method	Method Used in This Lab			Copy Available	Sample Frequency	
		Circle Appropriate Reference[1] Check next col. if copies available in lab.					
		Standard Method	ASTM	EPA		#/ Month	Peak Load
51. Cyanide, Total (mg/liter)	(a) Distillation & Silver Nitrate Titration	207A, 207B	D2036-74A	p. 40			
	(b) Distillation & Pyridine-Pyrazolone (or Pyridine - Barbituric Acid) Colorimetric	207A, 207C	D2036-74A	p. 40			
52. Fluoride (mg/liter)	(a) Distillation-SPADNS	121A, 121C	D1179-72A	p. 59			
	(b) Automated Complexone Method			p. 61			
	(c) Fluoride Electrode			p. 65			
53. Chlorine, Total Residual (mg/liter)	(a) Starch-Iodide Titration	204A	D1427-68A	p. 35			
	(b) Amperometric Titration	204A	D1427-68B	p. 35			
54. Oil and Grease (mg/liter)	(a) Gravimetric (Separatory Funnel Extraction)	137		p. 229			
	(b) Infrared (Separatory Funnel Extraction)			p. 232			
55. Phenols (mg/liter)	(a) Colorimetric (4-AAP Method with Distillation)	222E	D1783-70	p. 241			

Test and Unit	Method	Method Used in This Lab			Copy Available	Sample Frequency	
		Circle Appropriate Reference[1] Check next col. if copies available in lab.					
		Standard Method	ASTM	EPA		# / Month	Peak Load
56. Surfactants (mg/liter)	(a) Methylene Blue Colorimetric	159A	D2330-68	p. 157			
57. Algicides (mg/liter)	(a) Gas Chromatography			§			
58. Benzidine (mg/liter)	(a) Diazotization & Colorimetric			†			
59. Chlorinated Organic Compounds (Except Pesticides) (mg/liter)	(a) Gas Chromatography			§			
60. Pesticides (mg/liter)	(a) Gas Chromatography			§			
	(b) Thin Layer Chromatography			§			

CHART C. TABLE OF ANALYTICAL METHODS

Lab Name: ____________

Test and Unit	Method	Method Used in This Lab: Circle Appropriate Reference[1] Check next col. if copies available in lab. Standard Method	ASTM	EPA	Copy Available	Sample Frequency #/Month	Sample Frequency Peak Load
Physical and Biological Tests							
61. Color	(a) Platinum-cobalt Colorimetric	118		p. 36			
	(b) Spectrophotometric (Dominant wavelength, hue, luminance, purity)	206A		p. 39			
62. Specific Conductance (mho/cm @ 25° C)	(a) Wheatstone Bridge	154	D1125-64	p. 275			
63. Turbidity (Jackson Units)	(a) Turbidimeter Method	163A	D1889-71 (Sect. 10-16)	p. 295			
64. Streptococci Bacteria, Fecal (number/100 ml)	(a) MPN	409A					
	(b) Membrane Filter	409B					
	(c) Plate Count	409C					
65. Coliform Bacteria, Fecal (number/100 ml)	(a) MPN	407C					
	(b) Membrane Filter	408B					
66. Coliform Bacteria, Total (number/100 ml)	(a) MPN	407A					
	(b) Membrane Filter	408A					

CHART C. TABLE OF ANALYTICAL METHODS

Lab Name: ____________________

Test and Unit	Method	Method Used in This Lab			Copy Available	Sample Frequency	
		Circle Appropriate Reference[1] Check next col. if copies available in lab.					
		Standard Method	ASTM	EPA		# / Month	Peak Load
Radiological Tests							
67. Alpha, Total (pCi/liter)	(a) Proportional Counter	302	D1943-66				
	(b) Scintillation Counter		D1943-66				
68. Alpha, Counting Error (pCi/liter)	(a) Proportional Counter	302	D3085-72T				
	(b) Scintillation Counter		D3085-72T				
69. Beta, Total (pCi/liter)	(a) Proportional Counter	302	D1890-66				
70. Beta Counting Error (pCi/liter)	(a) Proportional Counter	302	D3085-72T				
71. Radium, Total (pCi/liter)	(a) Proportional Counter	304	D2460-70				
	(b) Scintillation Counter	304,305	D2460-70				

CHART C. TABLE OF ANALYTICAL METHODS

Lab Name: ____________

Test and Unit	Method	Method Used in This Lab Circle Appropriate Reference[2] Check next col. if copies available in lab.			Copy Available	Sample Frequency	
		Standard Method	ASTM	EPA		# / Month	Peak Load
Tests for Other Characteristics							
72. Temperature	(a) Thermometer or Thermistor	162		p. 286			
73. pH	(a) Electrometric	144A	D1293-65	p. 239			

CHART C. TABLE OF ANALYTICAL METHODS

Lab Name: ______________

Test and Unit	Method	Method Used in This Lab			Copy Available	Sample Frequency	
		Circle Appropriate Reference Check next col. if copies available in lab.					
		Standard Method	ASTM	EPA		# / Month	Peak Load
Tests for Air Characteristics							
74. Sulphur Dioxide	(a) Pararosaniline Method $\left\{\begin{array}{l}\text{Manual}\\\text{Automated}\end{array}\right.$			b.22385-7 φ			
75. Suspended Particulates	(a) High Volume Method			b.22388-90 φ			
76. Carbon Monoxide	(a) Nondispersive Infrared Spectrometry			b.22391 φ			
77. Photochemical Oxidants (Ozone)	(a) Chemluminescence, Continuous			b.22392 φ			
78. Hydrocarbons (minus Methane)	(a) GC-FID			b.22394 φ			
79. Nitrogen Dioxide	(a) Arsenite 24-Hr Sampling Method Manual Automated			b.15175 ▽ 22396 φ			
	(b) Chemluminescence, Continuous			b.15177 ▽			

Chart C. Table of Analytical Methods

Lab Name: ____________________

Test and Unit	Method	Method Used in This Lab Give Method Number or Page Check next col. if copies available in lab.			Copy Available	Sample Frequency	
		Standard Method	ASTM	EPA		# / Month	Peak Load
Non-referenced Tests in Use							

ALTERNATE ANALYTICAL METHOD

Name of Laboratory ______

(a) # ______ Test: ______

(b) If this is a modification of a referenced method,

(1) Which referenced method (give manual name and pages)? ______

(2) Purpose of modification: ______

(3) Brief description of modification: ______

(4) Literature reference, if any: ______

(c) If this is an alternate method,

(1) Purpose of use of alternate method: ______

(2) Brief description of method: ______

(3) Literature reference, if any: ______

(d) Have you applied to EPA for approval of this procedure? (c.f., Federal Register, Vol. 38, No. 199, October 16, 1973, p. 28760) ______

REFERENCE MARKS IN CHART C

1 - Federal Register, Vol. 35, No. 199, October 16, 1973.

2 - EPA Methods Manual, 1971.

Ψ - An introduction to atomic absorption spectrophotometry and a general procedure for trace metal analysis by atomic absorption is given in EPA Manual, pp. 78-91.

§ - Interim procedures for algicides, chlorinated organic compounds, and pesticides obtained from the Environmental Monitoring and Support Laboratory, USEPA, Cincinnati, Ohio 45268.

† - Estimated by the method of M.A. El-Dib, "Colorimetric Determination of Aniline Derivatives in Natural Waters," Journal of the Association of Official Analytical Chemists, Vol. 54, No. 6, November, 1971, pp. 1383-1387.

▽ - Federal Register, Vol. 38, No. 110, June 8, 1973.

φ - Federal Register, Vol. 36, No. 228, November 25, 1971.

• - Without Ca reduction.

Lab Name ______________________

PART 5. ANALYTICAL INSTRUMENTS AND SPECIAL APPARATUS

(1) Complete Chart D indicating analytical instruments and special apparatus available in the laboratory. See complete list of equipment, by analytical method, in the Appendix.

Completed by ______________________ Date ______________________
NAME TITLE

INDEX OF ANALYTICAL INSTRUMENTS AND SPECIAL APPARATUS

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Special Microbiological Equipment

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42.	Membrane Filters	59
43.	Colony Counters	59
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Lab Name ____________________

CHART D. ANALYTICAL INSTRUMENTS AND SPECIAL APPARATUS

Identify the instruments and apparatus in use and in good working condition in your laboratory.

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
1. Technicon Autoanalyzer AAI Units ____					
AAII Units ____					
Samplers ____					

Manifolds for:

- ☐ Alkalinity
- ☐ Ammonia Nitrogen (Colorimetric Phenate)
- ☐ Kjeldahl Nitrogen (Colorimetric Phenate)
- ☐ Kjeldahl Nitrogen (Selenium Method)
- ☐ Nitrate-Nitrite (Cd Reduction)
- ☐ Total Phosphorus or Orthophosphate
- ☐ Total Hardness
- ☐ Mercury (Cold Vapor Technique)
- ☐ Sulphur (Chloranilate)
- ☐ Chloride (Ferricyanide)
- ☐ Fluoride (Complexone)
- ☐ Phenols (4-AAP)
- ☐ Others (Specify):

____

____

Analytical Cartridges for:

- ☐ Ammonia Nitrogen (Colorimetric Phenate)
- ☐ Total Phosphorus or Orthophosphate
- ☐ Nitrate-Nitrite (Cd Reduction)
- ☐ Others (Specify):

____

____

Special Radiological Equipment

45. Alpha/Beta Particle Counters 61
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Special Air Equipment

48. Sulfur Dioxide Monitoring 63
49. High Volume Sampler (Particulates) 63
50. Carbon Monoxide Monitor 63
51. Total Hydrocarbons Monitor 64
52. Photochemical Oxidants Monitor 64
53. Nitrogen Dioxide Monitor 64
54. Other Air Monitoring Equipment Including Calibration Equipment 65

Accessory	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
Colorimeters ____					

Cells, tubular flow (Give number of each type.)

☐ 15 mm

☐ 50 mm

Filters:

Wavelength of Max. Transmittance

Wavelength of Max. Transmittance

Accessory	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
Recorders ____					

☐ Range Expansion

☐ Digital Printer

Associated Apparatus:

☐ Continuous Filter
☐ Proportioning Pump
☐ Planetary Pump
☐ Vapor-liquid Separator (for Hg Cold Vapor)
☐ Continuous Digester
☐ Others (Specify):

____

☐ Heating Bath
☐ 45° - 80° C Range
☐ With Distillation Coil & Head
☐ With Double Delay Coil
☐ High Temperature with 2 Distillation Coils

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
2. Colorimeters/Filter Photometers Range: ______					
Range: ______					

Filters:

Wavelength of Max. Transmittance	Bandwidth

Wavelength of Max. Transmittance	Bandwidth

Special Associated Apparatus (Specify): ______

______ ______

______ ______

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
3. Spectrophotometers (UV - visible) Recording (Range:) ______					
Manual (Range:) ______					

Special Attachments (Specify): ______

______ ______

______ ______

Metal	Lamps			Fuels				
	Hollow Cathode	Electric Discharge	Other (Specify)	Acetylene	Air	Nitrous Oxide	Argon	Hydrogen
Magnesium								
Manganese								
Mercury (Cold Vapor)								
Molybdenum								
Nickel								
Potassium								
Selenium (Gaseous Hydride)								
Silver								
Sodium								
Thallium								
Tin								
Titanium								
Vanadium								
Zinc								

Associated Equipment:

☐ Spectrophotometric gas cells, 10 cm, quartz windows (for Hg Cold Vapor)

☐ Mercury Cold Vapor Setup

☐ Others (Specify):

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
4. Atomic Absorption Spectrophotometers					

Recorders (Specify): ____________

____________ ____________

____________ ____________

Indicate lamps and fuels used for each metal:

Metal	Lamps			Fuels				
	Hollow Cathode	Electric Discharge	Other (Specify)	Acetylene	Air	Nitrous Oxide	Argon	Hydrogen
Aluminum								
Antimony								
Arsenic (Gaseous Hydride)								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium VI								
Chromium, Total								
Cobalt								
Copper								
Iron								
Lead								

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
5. Mercury Analyzers Technique:					
Range:					
Sensitivity:					

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
6. Flame Photometers Direct Reading:					
Internal Standard:					

Special Apparatus:

☐ Setup for Na in low-solids water (Air blower & filter, Oxy-hydrogen flame, polyethylene, or Teflon apparatus)

☐ Others (Specify):

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
7. Infrared Spectrophotometers Single Beam (Range:)					
Double Beam (Range:)					

Special Features:

IR Cells (Specify):

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
8. Conductivity Meters					
Field (Cell Type:)					
Laboratory (Cell Type:)					
Associated Apparatus (Specify):					

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
9. Electrometric Apparatus					
Electrometers:					
Field - ASTM Type I					
ASTM Type II					
Laboratory - ASTM I					
ASTM II					

Electrodes:	Manufacturer	Type
pH		
Dissolved Oxygen		
Ammonia		
Fluoride		
Cyanide		
Other (Specify):		

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
10. Automatic Titrimeters					

Recorders (Specify): ____________________

Frequently Used Electrodes (Specify): ____________________

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
11. Amperometric Titration Apparatus					

Instrument		# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
12. Analytic Balances						
Capacity	Sensitivity					

Certified Weights ☐ Certification ____________________

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
13. Carbon Analyzers Infrared (as CO_2) ____________					
Flame Ionization (as CH_4) ____________					

Instrument	# Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
14. Nephelometers/ Turbidimeters Range: ____					
Sensitivity Below 1 NTU: ____					

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
15. Blenders ____					

Instruments	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
16. Vacuum Pumps Type: ____					
Type: ____					

Apparatus	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
17. Magnetic Stirrers With Heater ____					
____					
With Timer ____					
____					

18. Drying Ovens

☐ 98° C ☐ 103°-105° C ☐ 180° C

19. **Muffle Furnance**

☐ 550° C

20. **Hot Plate (persulphate digestion)** ☐

Autoclave (persulphate digestion) ☐

21. **Water Baths/Incubators**

☐ 10°-15° C

☐ 25° C with rack (for conductance measurements)

☐ 100° C, well stirred, with Neoprene coated wire rack for 40-50 ml sample tubes (for Brucine Nitrate Method)

☐ 20° C incubator (for B.O.D.) with circulator

22. **B.O.D. Incubation Bottles** **Number** ____________

23. **Gravimetric Evaporating/Weighing Dishes**	**Number**
Porcelain	____________
Vycor	____________
Platinum	____________

Apparatus	Number
24. Dessicators Type: ______	
Type: ______	

Apparatus	Number
25. Kjeldahl Distillation Apparatus Macro ______	
Micro ______	

Apparatus	Number
26. Arsine Generator & Absorption Apparatus ______	

Apparatus	Number
27. Cyanide Distillation Apparatus ______	

Apparatus	Number
28. Soxhlet Extraction Apparatus Thimble Size: ______	

Apparatus	Number
29. Phenol Distillation Setups ______	

Apparatus	Number
30. Nessler Tubes, matched sets, APHA standard	
50 ml, tall ______	
100 ml, tall ______	

Apparatus	Model	Temperature	Cubic Feet
31. Refrigerators			

Apparatus	Certification	Range
32. Special Thermometers		

Apparatus
33. **Thin-Layer Chromatography Apparatus** (Describe chambers; plates, commercial or homemade; spray reagents and apparatus; spotting apparatus; special equipment)
______
______
______
______
______
______

34. **Column Chromatography Apparatus** (Describe columns; adsorbents - type, source, grade, special handling; solvent evaporation apparatus; special equipment)

35. **Gas Chromatographs** (Describe for each instrument: make and model; column type-capillary, 1/8 in., 1/4 in., etc., temperature programming; detector type and model; recorder; most commonly used columns; special equipment)

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
36. Other Special Instruments (Mass Spec., NMR, Flowmeters, Electrom Microscope, etc.)					

CHART D. ANALYTICAL INSTRUMENTS AND SPECIAL APPARATUS

Lab Name: ____________________

Special Microbiological Equipment

37. **Incubation Oven** 35 ± 0.5° C ☐

☐ Humidity controlled? (Specify relative humidity) ____________________

38. **Water Baths**

☐ 35 ± 0.5° C ☐ 44.5 ± 0.2° C

39. **Autoclave** (to 121° C) ☐

	# of Units	Manufacturer	Magnification	Light Source
40. Light Microscope Type: ____________				
Type: ____________				

	Plastic	Glass	Other (Specify)
41. Miscellaneous Microbiological Containers			
Sample Bottles			
Inoculation Tubes			
Dilution Bottles			
Containers for Media			
Petri Dishes			
Other (Specify)			

	Manufacturer	Type
42. Membrane Filters		

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
43. Colony Counters					

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
44. Other Microbiological/Biological Instrumentation					

CHART D. ANALYTICAL INSTRUMENTS AND SPECIAL APPARATUS

Lab Name: ____________

Special Radiological Equipment

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
45. **Alpha & Beta Particle Counters** Windowless Gas-Flow Proportional Counter					
Thin Window Gas-Flow Proportional Counter					
Alpha Scintillation Counter					
Beta Scintillation Counter					
Liquid Scintillation Counter					

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
46. **Spectrometer Systems** Alpha Spectrometer (Surface Barrier Type)					
Detector					
Analyzer					

Other Pertinent Information ____________

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
46. Spectrometer Systems (Con't.)					
Gamma Spectrometer					
Detector					
Analyzer					
Other Pertinent Information					

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
47. Other Radiological Instrumentation (Radon Gas Counters, Survey Instruments, etc.)					

CHART D. ANALYTICAL INSTRUMENTS AND SPECIAL APPARATUS

Lab Name: ____________________

Special Air Monitoring Equipment

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
48. Sulphur Dioxide Monitor (Field Sampling/Lab Analysis) Field Sampler ____					
Lab Analytical Method: ____					
(Field Sampling/Field Analysis) Field Sampler/Analyzer ____					
Analytical Method: ____					

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
49. Suspended Particulates (High Volume Sampler) Filter Type ____					

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
50. Carbon Monoxide Monitor					

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
51. Total Hydrocarbons (corrected for CH_4) Monitor					

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
52. Photochemical Oxidants (O_3) Monitor					

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
53. Nitrogen Dioxide Monitor (Field Sampling/ Lab Analysis) Field Sampler					
Lab Analytical Method:					
(Field Sampling/ Field Analysis) Field Sampler/Analyzer					
Analytical Method:					

Instrument	# of Units	Manufacturer	Model	Year Purchased	Operating Manual Avail. in Lab.
54. Other Air Monitoring Equipment Including Calibration Equipment					
Permeation Tubes					
Standard Cylinders					
Gas Phase Titration-Commercial					
-Home Made					
Air Dilution Systems					
Variable Temperature Bath: 25° C ± 0.1° C					

INTERNAL AND EXTERNAL CONTROLS

The first three sections of this chart contain lists on which the availability of written operating procedures is to be checked. These parts cover instrument maintenance and calibration, all aspects of sampling, and the quality control program of the laboratory. You may be asked to show these documents to the evaluator during the onsite inspection and to discuss them with him.

This check list should not be looked upon as a demand for written procedures (for example, a Quality Control Program) in a particular standard format. The important thing is that the principal laboratory controls should be documented in a permanent way. Some procedures may be brief or may not include all of the items to be checked. In the list please check those items which you believe to be adequately documented. The onsite visit will provide an opportunity to discuss the completeness of the documentation with the evaluator.

Part 4 of this chart asks for information on participation in interlaboratory proficiency testing programs. Information is required on the test methods covered in any plan in which you have participated, the organization conducting the program and the date of the last check sample reported upon.

You will be rated on the extent of your participation in such programs. However, as of the present, the actual standing you have achieved in proficiency tests is not a part of the scoring system for this evaluation.

Lab Name ____________________

PART 6. INTERNAL AND EXTERNAL CONTROLS

CHART E

	Available	
	Yes	No
1. Control of Analytical Methods and Instruments		
(1) Written Instrument Maintenance and Calibration Procedures and Log Books		
(2) Written Bench Operating Procedures		
2. Control of Sampling and Sample Preservation		
(1) Written Sampling Procedures Covering:		
Sampling Plans and Sampling Equipment		
Sample Collection and Preservation		
Identification and Storage of Samples		
Laboratory Handling of Samples (Request for analysis, sample preparation, timely performance, etc.)		
(2) Written Description of the Chain of Custody of Samples		
(3) Written Procedures for Field Measurement (Flow, critical tests: D.O., Residual C1, etc.)		
(4) Written Procedures for Monitoring (Water supply, effluents, ambient air, stacks, mobile vehicles, pesticides, radiation, etc.)		
3. Quality Control		
(1) Written Quality Control Program Covering:		
Quality Policy		
Assignment of Responsibility		
Training in Quality Control Methods		
Control of Purchased Chemicals/Reagents		
Internal Field and Laboratory Checks:		
Precision/Accuracy		
Routine Duplicates, Spiked, and Standard Samples		
Statistical Methods, Including Control Charts and/or Computer Methods		
(2) Written Description of Lab Record System (Data handling/calculations, data review, validation and audit)		
(3) Written Description of Lab Report Systems		

(4) If you have a Quality Control Manual, please provide a copy. Attached ☐

4. Inter-laboratory Proficiency Testing Programs

Test Method	Participated in Program of:					Date of Last Check Sample	Within Acceptable Limits
	EPA	CDC	State	USGS	Other (Specify)		

Completed by: ______________________ Date ______________

NAME TITLE

HANDOUT 3

METHOD OF ANALYSIS BREAKDOWN

SAMPLE WORKSHEET
BREAKING FLUORIDE ANALYSIS (By Electrode) INTO WORK ELEMENTS

EPA Method Work Elements	Observed Work Elements	Noted Differences
1. Calibrate Electrometer		
2. Place 50 ml of sample and 50 ml(1) of buffer in a 150 ml beaker.		
3. Place magnetic stirrer in beaker and stir while inserting electrodes.		
4. Wait at least three (3) minutes to allow electrode reading to stabilize.		
5. Record potential (mv) reading in notebook.		
6. Use calibration chart to convert potential (mv) reading to fluoride concentration and record in notebook.		

Note

(1) Highly basic samples (7pH11) will need additional acid to adjust pH to 8.3

Outline of the Initial Meeting

I. Attendees

  A. Laboratory Officers
  B. Laboratory Director
  C. Lower Level People Not Required

II. Opening Statement

  A. Purpose of Inspection
  B. Objectives
  C. Process
  D. Expected Results
  E. Time Table
  F. Questions on the Part of the Laboratory

III. General Questions to the Laboratory

  A. Is there a Laboratory Methods Manual?
  B. Is there a Laboratory Quality Control Manual?
  C. Is there a Director of Quality Assurance?
  D. Questions from EPA Form 3560-3

    1. Are EPA-approved analytical methods used?
    2. If alternate methods are used, has regional approval been obtained for their use?
    3. What procedures are used to calibrate?
      a. Atomic Adsorption Spectrometer?
      b. Gas Chromatograph?
      c. pH Meters?
      d. Specific Ion Detectors?
    4. Have the laboratory describe their quality control procedure
    5. What percent of the laboratory sample load is duplicate samples?
    6. What percent of the laboratory sample load is spiked?
    7. Is the laboratory certified by the State or by any other certifying body?

E. Are NBS Standard Reference Materials (SRM's) Used as a Routine Part of Calibration and Quality Control Programs?

F. What Happens to a Sample After It Enters the Laboratory?

IV. Permit-Specific Questions

A. What Methods are Used to Analyze Pollutant Parameters in the Plant Permit?

B. Are These EPA-Approved Methods?

1. If not, has approval been obtained from the EPA Region for their use?
2. If not, has the analytical method been validated by an interlaboratory collaborative test?

C. Name the People Who Perform the Analyses for the Chemical Found in the Plant Permit. (These people are to be visited during the laboratory tour.)

V. Laboratory Self-Appraisal Check List for Analytic Quality Control (use as suggested)

VI. Laboratory Tour

HANDOUT 5
(1 of 7)

LABORATORY SELF-APPRAISAL QUALITY CONTROL CHECK LIST

This check list was designed for laboratory self-evaluation, giving three descriptions to each facet of a quality control system. In all cases the "5" choice is the most desirable and effective mode of operation. "3" is marginal and tolerable. "1" is definitely unacceptable and ineffective as a mode of operation.

After all the applicable questions have been answered, a total score should be computed. Generally a total score of 120 or more may be considered as indicating good quality control. Scores between 85 and 119 indicate a need for improvement but laboratory data may be considered adequate. Scores below 85 indicate poor quality control; laboratory data quality may be in violation of the Discharge Permit and corrective action is needed immediately.

Score

1. Acceptable levels of quality of the laboratory's routine performance are:
   a. Clearly defined in writing for all characteristics. 5
   b. Are defined in writing for some characteristics and are dependent on experience, memory, and/or verbal communication. 3
   c. Only defined by experience and verbal communication. 1 ______

2. Criteria for acceptable level of quality of the laboratory's routine performance are obtained by:
   a. Monitoring laboratory performance in a structured program of inter- and intra-laboratory evaluation. 5
   b. Scientific determination of what is technically feasible. 3
   c. Laboratory determination of what can be done using currently available equipment, techniques and manpower. 1 ______

3. The quality control coordinator has the authority to:
   a. Affect the quality of analytical results by inserting controls to assure that the methods meet the user's needs for precision, accuracy, sensitivity, and specificity. 5
   b. Reject suspected results and halt any method that produces high levels of discrepancies. 3
   c. Submit suspected results to laboratory management for a decision on disposition. 1 ______

4. Accountability for quality is:
   a. Clearly defined for all laboratory supervisors where their actions have an impact on quality. 5
   b. Vested with the quality control coordinator who must use whatever means possible to achieve quality goals. 3
   c. Not defined. 1 ______

5. Quality control is included in laboratory long-range planning and:
   a. Is considered an important factor with regard to changing user demands, new applications, legal considerations and technical advances in control and methods. 5
   b. Is considered part of the technology or service. 3
   c. Not considered a factor for planning purposes. 1 ______

6. Measuring, gauging and analytical instruments are
   a. Maintained operative, accurate and precise by regular checks and calibrations against stable standards which are traceable to the U. S. Bureau of Standards. 5
   b. Periodically checked against a zero point or other reference and are examined for evidence of physical damage, wear, or inadequate maintenance. 3
   c. Checked only when they stop working or when excessive defects are experienced which can be traced to inadequate instrumentation. 1 ________

7. Reagents and chemicals (critical items) are:
   a. Procured from suppliers who must submit samples for test and approval prior to initial shipment. 5
   b. Procured from suppliers who certify they can meet all applicable specifications. 3
   c. Procured from suppliers on the basis of price and delivery only. 1 ________

8. Analytical reagents and chemicals are:
   a. Checked 100% against specification and quantity and for certification where required and accepted only if they conform to all specifications. 5
   b. Spot-checked for proper quantity and for shipping damage. 3
   c. Released to analyst by the receiving clerk without being checked as above. 1 ________

9. Information on off-spec purchased materials is:
   a. Transmitted to the supplier with a request for corrective action. 5
   b. Filed for future use. 3
   c. Not maintained. 1 ________

10. Off-spec purchased materials are:
   a. Submitted to a review by Quality Control Coordinator and Chief Chemist for disposition. 5
   b. Submitted to a Service Section for determination on acceptability. 3
   c. Used because of scheduling requirements. 1 ________

11. Reagent inventories are maintained on:
   a. First-in, first-out basis. 5
   b. Random selection in stock room. 3
   c. Last-in, first-out basis. 1 ________

12. Reagent inventories are:
   a. Identified as to type, age, and acceptance status. 5
   b. Identified as to material only. 3
   c. Not identified in writing. 1 ________

13. Reagents and chemicals that have limited self life are:
    a. Identified as to shelf life expiration date and systematically issued from stock only if they are still within that date. 5
    b. Issued on a first-in, first-out basis, expecting that there is enough safety factor so that the expiration date is rarely exceeded. 3
    c. Issued at random from stock. 1 ________

14. The operating conditions of the analytical methods are:
    a. Clearly defined in writing in the method for each significant variable. 5
    b. Controlled by supervision based on general guidelines. 3
    c. Left up to the bench chemist. 1 ________

15. Analytical methods are checked:
    a. During the analysis for conformance to operating conditions and to specifications. 5
    b. After the analysis to determine acceptability of the results. 3
    c. Not at all. 1 ________

16. Revisions to laboratory technical documents are:
    a. Clearly spelled out in written form and distributed to all parties affected on a controlled basis (which assures that the change will be implemented and permanent). 5
    b. Communicated through memoranda to key people who are responsible for implementing the change through whatever method they choose. 3
    c. Communicated verbally to operating personnel. 1 ________

17. Changes to methods and other technical documents are:
    a. Analyzed to make sure that any harmful side effects are known and controlled prior to implementing the revision. 5
    b. Installed on a trial or gradual basis, monitoring the product to see if the revision has a net beneficial effect. 3
    c. Installed immediately with action for correcting side effects taken if they show up in the final results. 1 ________

18. Revisions to technical documents are:
    a. Recorded as to date, serial number, etc. when the revision becomes effective. 5
    b. Recorded as to the date the revision was made on written specifications. 3
    c. Not recorded with any degree of precision. 1 ________

19. The capability of a given analytical method to produce results within specification limit is:
    a. Known through method capability analysis (EPA QA Charts) to be able to produce consistently acceptable results. 5
    b. Assumed to be able to produce a reasonably acceptable result. 3
    c. Unknown. 1 ________

20. Noted discrepancies in analytical methods are:
    a. Analyzed immediately to seek out the causes and apply corrective action. 5
    b. Checked out when time permits. 3
    c. Not detectable with present controls and procedures. 1 ________

21. Decisions on acceptability of questionable results are made by:
    a. A review group consisting of the chief chemist, quality control coordinator and others who can render expert judgment. 5
    b. An informal assessment by quality control. 3
    c. The bench chemist. 1 ________

22. Final acceptance of the results is made:
    a. By replicating statistically adequate samples. 5
    b. By routine acceptance because of lack of complaints. 3
    c. On faith. 1 ________

23. Follow-up action is taken to:
    a. Identify assignable causes for suspected determinations. 5
    b. Make sure that method errors have been corrected. 3
    c. Not taken. 1 ________

24. Data reports on quality are distributed to:
    a. All levels of management. 5
    b. To one level of management only. 3
    c. Quality control only. 1 ________

25. Product Quality reports contain:
    a. Information on trends, required action, and danger areas. 5
    b. Information on suspected analyses and their causes. 3
    c. Number of analyses per month. 1 ________

26. Quality analysis is performed in order to:
    a. Seek out the optimum levels of operations. 5
    b. Provide for highlighting trouble spots. 3
    c. Fix the blame for sub-standard results. 1 ________

27. When key personnel changes occur:
    a. Specialized knowledge and skills are retained in the form of documented methods and descriptions. 5
    b. Replacement people can acquire the knowledge of their predecessors from co-workers, supervisors, and detailed study of specifications and memoranda. 3
    c. Knowledge is lost and must be regained through long experience or trial-and-error. 1 ________

28. The people who affect the quality performance of bench chemists, supervisors, etc. are:
    a. Trained in standards of quality and the methods by which high quality can be achieved. 5
    b. Told about quality only when their work falls below acceptable levels. 3
    c. Are reprimanded when quality deficiencies are directly traceable to the chemists' analytical work. 1 ________

29. Training of new employees is accomplished by:
    a. A programmed system of training where elements of training, including quality standards, are included in a training checklist. The chemist's work is immediately re-checked by supervisors for errors or defects and the information is fed back instantaneously for corrective action. 5
    b. On-the-job training by the supervisor who gives an overview of quality standards are learned as normal results are fed back to the chemist. 3
    c. On-the-job learning with training on the rudiments of the job by senior co-workers. 1 ________

30. Auditing of the quality control department is:
    a. Performed on a random but regular basis in order to verify that all quality procedures are being implemented and are effective. 5
    b. Performed whenever a suspicion arises that there are areas of ineffective performance. 3
    c. Never performed. 1 ________

31. Corrective action to reduce failure costs is:
    a. An ongoing program with specific objectives, measurements, and target dates. 5
    b. Taken when deficient results threaten schedules. 3
    c. Taken after considerable losses have occurred in the laboratory or in the field. 1 ________

32. The Management through the Proficiency Analytical Testing (PAT) Program, regards the laboratory's performance quality as:
    a. Significantly better than peer laboratories. 5
    b. About the same as peer laboratories. 3
    c. Significantly worse than peer laboratories or not done. 1 ________

33. Support for laboratory quality objectives, goals, and results by top management is indicated by:
    a. A clear statement of quality objectives by the top executive with continuing visible evidence of sincerity to all levels of the organization. 5
    b. Periodic meetings among the section heads of service, research and development, and quality assurance on quality objectives and progress toward their achievement. 3
    c. A "one-shot" statement of the desire for product quality by the top executive after which the quality control staff is on its own. 1 ________

34. The quality control system is:
    a. Formalized and documented by a set of procedures which clearly describe the activities necessary and sufficient to achieve desired quality objectives for initial design through to final delivery to the user. 5
    b. Contained in methods procedures or is implicit in those procedures. Experience with the materials, product and equipment is needed for continuity of control. 3
    c. Undefined in any procedures and is left to the current managers or supervisors to determine as the situation dictates. 1 ________

SUMMARY

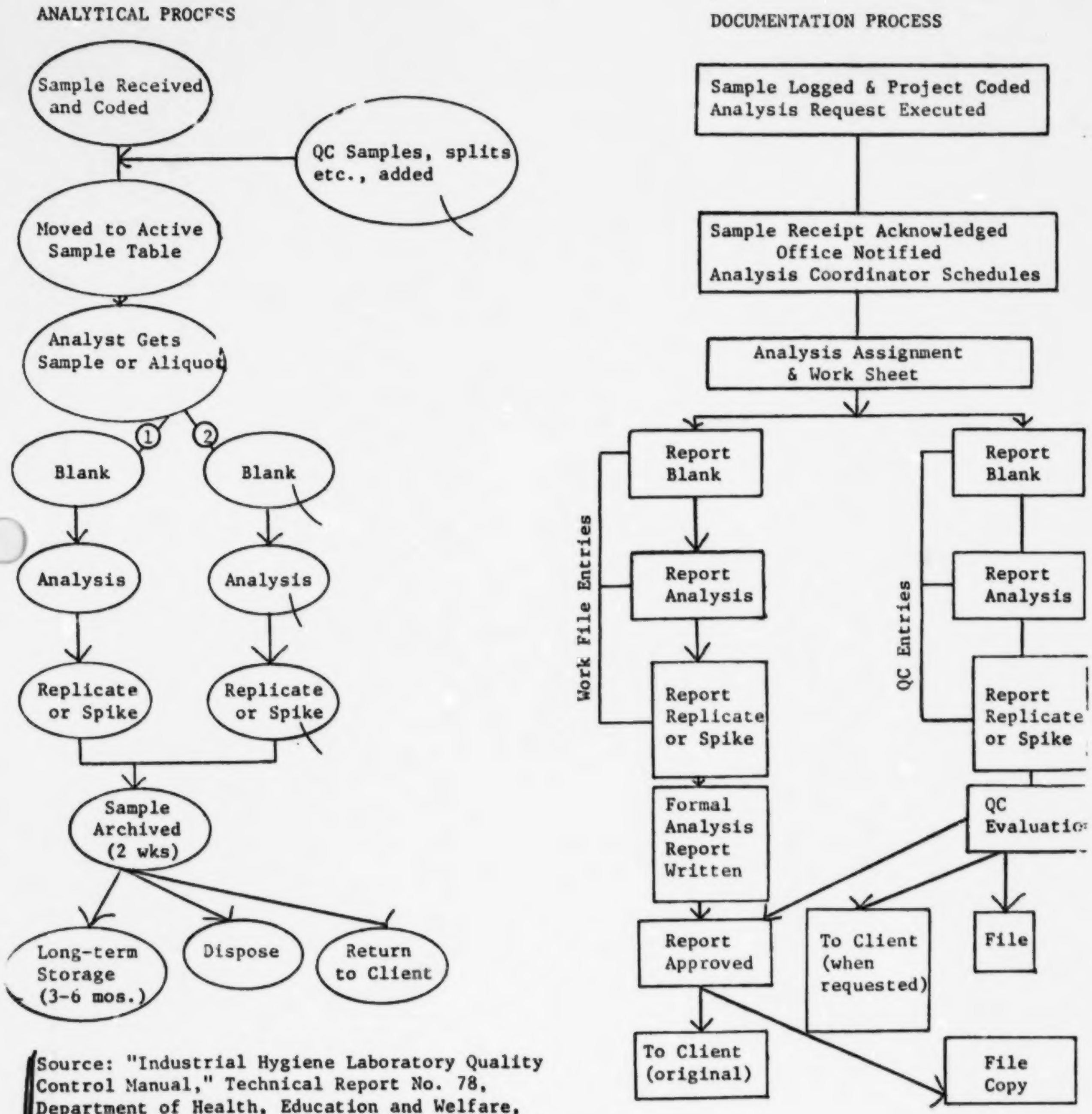
Strong Points:

Weak Points:

Improvement Goals:

HANDOUT 6

SAMPLE CONTROL SYSTEM


ANALYTICAL PROCESS
DOCUMENTATION PROCESS
Sample Received and Coded
QC Samples, splits etc., added
Moved to Active Sample Table
Analyst Gets Sample or Aliquot
①
②
Blank
Blank
Analysis
Analysis
Replicate or Spike
Replicate or Spike
Sample Archived (2 wks)
Long-term Storage (3-6 mos.)
Dispose
Return to Client
Sample Logged & Project Coded Analysis Request Executed
Sample Receipt Acknowledged Office Notified Analysis Coordinator Schedules
Analysis Assignment & Work Sheet
Work File Entries
Report Blank
Report Analysis
Report Replicate or Spike
Formal Analysis Report Written
Report Approved
To Client (original)
File Copy
QC Entries
Report Blank
Report Analysis
Report Replicate or Spike
QC Evaluatio
To Client (when requested)
File

Source: "Industrial Hygiene Laboratory Quality Control Manual," Technical Report No. 78, Department of Health, Education and Welfare, National Institute for Occupational Safety and Health, Division of Physical Sciences and Engineering, Cincinnati, Ohio 1976

SYMBOLS ○ Analytical Procedure ☐ Documentation Procedure

Q Quality Control Procedure

HANDOUT 7

SAMPLE CONTROL FORM

SAMPLE STORAGE, RETENTION & RELEASE

TO: ____________________

____

____

FROM: ____________________

____

____

Sample Description: ____

____

____

____

Project Code: ______________ Date Sample Received: ____________________

Sample No. (s): ____________________

Sample to be retained by lab: ____________________

Sample to be returned to customer: ______________

Sample portion to be retained by lab, remainder to Customer:

____

Special precautions for handling (combustible? detrimental to health? etc.)

____

____

____

Sample delivered to lab by: ____________________

Sample received at lab by: ____________________

Sample returned to Customer (date): ____________________

Signature of person picking up sample: ____________________

Signature of employee releasing sample: ____________________

Source: "Industrial Hygiene Laboratory Quality Control Manual", Technical Report No. 78, Department of Health Education and Welfare, National Institute for Occupational Safety and Health, Division of Physical Sciences and Engineering, Cincinatti, Ohio (1976)

STANDARD ANALYTICAL TEST

(A) 40 CFR Part 136

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PART 136—GUIDELINES ESTABLISHING TEST PROCEDURES FOR THE ANALYSIS OF POLLUTANTS

Sec.

136.1 Applicability.
136.2 Definitions.
136.3 Identification of test procedures.
136.4 Application for alternate test procedures.
136.5 Approval of alternate test procedures.

AUTHORITY: Sec. 304(g) of Federal Water Pollution Control Act Amendments of 1972 86 Stat. 816, et seq., Pub. L. 92-500).

§ 136.1 Applicability.

The procedures prescribed herein shall, except as noted in § 136.5, be used to perform the measurements indicated whenever the waste constituent specified is required to be measured for:

(a) An application submitted to the Administrator, or to a State having an approved NPDES program, for a permit under section 402 of the Federal Water Pollution Control Act as amended (FWPCA), and,

(b) Reports required to be submitted by dischargers under the NPDES established by Parts 124 and 125 of this chapter, and,

(c) Certifications issued by States pursuant to section 401 of the FWPCA, as amended.

[38 FR 28758, Oct. 16, 1973]

§ 136.2 Definitions.

As used in this part, the term:

(a) "Act" means the Federal Water Pollution Control Act, as amended, 33 U.S.C. 1314, et seq.

(b) "Administrator" means the Administrator of the U.S. Environmental Protection Agency.

(c) "Regional Administrator" means one of the EPA Regional Administrators.

(d) "Director" means the Director of the State Agency authorized to carry out an approved National Pollutant Discharge Elimination System Program under section 402 of the Act.

(e) "National Pollutant Discharge Elimination System (NPDES)" means the national system for the issuance of permits under section 402 of the Act and includes any State or interstate program which has been approved by the Admin-

TABLE I.—*List of approved test procedures*[1]—Continued

Parameter and units	Method	1974 EPA methods[1]	14th ed. standard methods	References (page nos.)		Other approved methods
				Pt. 31 1975 ASTM	USGS methods	
BACTERIA—Continued						
7. Coliform (total)[4] in presence of chlorine, number per 100 ml.	MPN;[6] membrane filter with enrichment.		916			
			933			
8. Fecal streptococci,[4] number per 100 ml.	MPN;[4] membrane filter; plate count.		943			
			944		[7] (50)	
			947			
9. Benzidine, milligrams per liter.	Oxidation—colorimetric.[9]					
10. Biochemical oxygen demand, 5-d (BOD5), milligrams per liter.	Winkler (Azide modification) or electrode method.		543		[7] (50)	
						[16] (17)
11. Bromide, milligrams per liter.	Titrimetric, iodine-iodate.	14		323	58	
12. Chemical oxygen demand (COD), milligrams per liter.	Dichromate reflux.	20	550	472	124	[8] (610)
						[10] (17)
13. Chloride, milligrams per liter.	Silver nitrate; mercuric nitrate; or automated colorimetric-ferricyanide.		303	297		
		29	304	265		[8] (615)
		31	613		[11] (46)	
14. Chlorinated organic compounds (except pesticides), milligrams per liter.	Gas chromatography [12]					
15. Chlorine—total residual, milligrams per liter.	Iodometric titration, amperometric or starch-iodine end-point; DPD colorimetric or Titrimetric methods (these last 2 are interim methods pending laboratory testing).		318			
		35	322	278		
			332			
			329			
16. Color, platinum cobalt units or dominant wave length, hue, luminance, purity.	Colorimetric; spectrophotometric; or ADMI procedure.[13]	36	64		52	
		39	66			
17. Cyanide, total,[14] milligrams per liter.	Distillation followed by silver nitrate titration or pyridine pyrazolone (or barbituric acid) colorimetric.	40	361	503	85	[10] (22)
18. Cyanide amenable to chlorination, milligrams per liter.	...do.	49	370	505		
19. Dissolved oxygen, milligrams per liter.	Winkler (Azide modification) or electrode method.	51	443	363	126	[8] (609)
		56	450			
20. Fluoride, milligrams per liter.	Distillation[6] followed by ion electrode; SPADNS; or automated complexone.		389			
		65	391	307	93	
		59	393	305		
		61	614			
21. Hardness—Total, as $CaCO_3$, milligrams per liter.	EDTA titration; automated colorimetric; or atomic absorption (sum of Ca and Mg as their respective carbonates).	68	202	161	94	[8] (617)
		70				
22. Hydrogen ion (pH), pH units.	Electrometric measurement.	239	460	178	129	[8] (606)
23. Kjeldahl nitrogen (as N), milligrams per liter.	Digestion and distillation followed by nesslerization, titration, or electrode; automated digestion automated phenolate.	175	437		122	[8] (612)
		165				
		182				
METALS						
24. Aluminum—Total, milligrams per liter.	Digestion[15] followed by atomic absorption[16] or by colorimetric (Eriochrome Cyanine R).	92	152		[11] (19)	
			171			
25. Aluminum—Dissolved, milligrams per liter.	0.45 micron filtration[17] followed by referenced methods for total aluminum.					
26. Antimony—Total, milligrams per liter.	Digestion[15] followed by atomic absorption.[16]	94				
27. Antimony—Dissolved, milligrams per liter.	0.45 micron filtration[17] followed by referenced method for total antimony.					
28. Arsenic—Total, milligrams per liter.	Digestion followed by silver diethyldithiocarbamate; or atomic absorption.[15] [16]		283			
		9	283		[11] (31)	
		95	159		[11] (37)	

See footnotes at end of table.

HANDOUT 8
(4 of 10)

TABLE I.—*List of approved test procedures*[1]—Continued

Parameter and units	Method	1974 EPA methods	14th ed. standard methods	References (page nos.)		Other approved methods
				Pt. 31 1975 ASTM	USGS methods [3]	
METALS—Continued						
29. Arsenic—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total arsenic.					
30. Barium—Total, milligrams per liter.	Digestion [14] followed by atomic absorption.[15]	97	152		82	
31. Barium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total barium.					
32. Beryllium—Total, milligrams per liter.	Digestion [14] followed by atomic absorption [15] or by colorimetric (Aluminon).	99	152 177		53	
33. Beryllium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total beryllium.					
34. Boron—Total, milligrams per liter.	Colorimetric (Curcumin)	13	287			
35. Boron—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total boron.					
36. Cadmium—Total, milligrams per liter.	Digestion [14] followed by atomic absorption [15] or by colorimetric (Dithizone).	101	148 152	345	62	[5] (619); [16] (37)
37. Cadmium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total cadmium.					
38. Calcium—Total, milligrams per liter.	Digestion [14] followed by atomic absorption; or EDTA titration.	103	148 189	345	66	
39. Calcium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total calcium.					
40. Chromium VI, milligrams per liter.	Extraction and atomic absorption: colorimetric (Diphenylcarbazide).	89, 105	 192		76 75	
41. Chromium VI—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for chromium VI.					
42. Chromium—Total, milligrams per liter.	Digestion [14] followed by atomic absorption [15] or by colorimetric (Diphenylcarbazide).	105	148 192	345 286	78 77	[5] (619)
43. Chromium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total chromium.					
44. Cobalt—Total, milligrams per liter.	Digestion [14] followed by atomic absorption.[15]	107	148	345	80	[16] (37)
45. Cobalt—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total cobalt.					
46. Copper—Total, milligrams per liter.	Digestion [14] followed by atomic absorption [15] or by colorimetric (Neocuproine).	108	148 196	345 293	53	(619) [16] (37)
47. Copper—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total copper.					
48. Gold—Total, milligrams per liter.	Digestion [14] followed by atomic absorption.[15]					
49. Iridium—Total, milligrams per liter.	Digestion [14] followed by atomic absorption.[15]					
50. Iron—Total, milligrams per liter.	Digestion [14] followed by atomic absorption [15] or by colorimetric (Phenanthroline).	110	148 208	345 326	102	[5] (619)
51. Iron—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total iron.					
52. Lead—Total, milligrams per liter.	Digestion [14] followed by atomic absorption [15] or by colorimetric (Dithizone).	112	148 215	345	105	[5] (619)

See footnotes at end of table.

TABLE I.—*List of approved test procedures*[1]—Continued

Parameter and units	Method	1974 EPA methods	14th ed. standard methods	References (page nos.)		Other approved methods
				Pt. 31 1975 ASTM	USGS methods[9]	
METALS—Continued						
53. Lead—Dissolved, milligrams per liter.	0.45 micron filtration[17] followed by referenced method for total lead.					
54. Magnesium—Total, milligrams per liter.	Digestion[13] followed by atomic absorption; or gravimetric.	114	118 221	315	100	[3] (619)
55. Magnesium—Dissolved milligrams per liter.	0.45 micron filtration[17] followed by referenced method for total magnesium.					
56. Manganese—Total milligrams per liter.	Digestion[13] followed by atomic absorption[18] or by colorimetric (Persulfate or periodate).	116	118 225, 227	345	111	[3] (619)
57. Manganese—Dissolved milligrams per liter.	0.45 micron filtration[17] followed by referenced method for total manganese.					
58. Mercury—Total, milligrams per liter.	Flameless atomic absorption.	118	156	338	[11] (51)	
59. Mercury—Dissolved, milligrams per liter.	0.45 micron filtration[17] followed by referenced method for total mercury.					
60. Molybdenum—Total, milligrams per liter.	Digestion[13] followed by atomic absorption.[18]	139		350		
61. Molybdenum—Dissolved, milligrams per liter.	0.45 micron filtration[17] followed by referenced method for total molybdenum.					
62. Nickel—Total, milligrams per liter.	Digestion[13] followed by atomic absorption[18] or by colorimetric (Heptoxime).	141 223	118	345	115	
63. Nickel—Dissolved, milligrams per liter.	0.45 micron filtration[17] followed by referenced method for total nickel.					
64. Osmium—Total, milligrams per liter.	Digestion[13] followed by atomic absorption.[18]					
65. Palladium—Total, milligrams per liter.	Digestion[13] followed by atomic absorption.[18]					
66. Platinum—Total, milligrams per liter.	Digestion[13] followed by atomic absorption.[18]					
67. Potassium—Total, milligrams per liter.	Digestion[13] followed by atomic absorption, colorimetric (Cobaltinitrite), or by flame photometric.	143	235 234	403	134	[3] (620)
68. Potassium—Dissolved, milligrams per liter.	0.45 micron filtration[17] followed by referenced method for total potassium.					
69. Rhodium—Total, milligrams per liter.	Digestion[13] followed by atomic absorption.[18]					
70. Ruthenium—Total, milligrams per liter.	Digestion[13] followed by atomic absorption.[18]					
71. Selenium—Total, milligrams per liter.	Digestion[13] followed by atomic absorption.[18] [19]	145	159			
72. Selenium—Dissolved, milligrams per liter.	0.45 micron filtration[17] followed by referenced method for total selenium.					
73. Silica—Dissolved, milligrams per liter.	0.45 micron filtration[17] followed by colorimetric (Molybdosilicate).	274	487	398	139	
74. Silver—Total,[20] milligrams per liter.	Digestion[13] followed by atomic absorption[18] or by colorimetric (Dithizone).	146	148 213		142	[3](619) [10](37)
75. Silver—Dissolved,[20] milligrams per liter.	0.45 micron filtration[17] followed by referenced method for total silver.					
76. Sodium—Total, milligrams per liter.	Digestion[13] followed by atomic absorption or by flame photometric.	147	250	403	143	[3] (621)
77. Sodium—Dissolved, milligrams per liter.	0.45 micron filtration[17] followed by referenced method for total sodium.					

See footnotes at end of table.

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TABLE I.—*List of approved test procedures*[1]—Continued

Parameter and units	Method	References (page nos.) 1974 EPA methods	References (page nos.) 14th ed. standard methods	References (page nos.) Pt. 31 1975 ASTM	References (page nos.) USGS methods[2]	Other approved methods
METALS—Continued						
78. Thallium—Total, milligrams per liter.	Digestion[15] followed by atomic absorption.[16]	149				
79. Thallium—Dissolved, milligrams per liter.	0.45 micron filtration[17] followed by referenced method for total thallium.					
80. Tin—Total, milligrams per liter.	Digestion[15] followed by atomic absorption.[16]	150			[11] (63)	
81. Tin—Dissolved, milligrams per liter.	0.45 micron filtration[17] followed by referenced method for total tin.					
82. Titanium—Total, milligrams per liter.	Digestion[15] followed by atomic absorption.[16]	151				
83. Titanium—Dissolved, milligrams per liter.	0.45 micron filtration[17] followed by referenced method for total titanium.					
84. Vanadium—Total, milligrams per liter.	Digestion[15] followed by atomic absorption[16] or by colorimetric (Gallic acid).	153 ……	152 250	 441	 [11] (67)	
85. Vanadium—Dissolved, milligrams per liter.	0.45 micron filtration[17] followed by referenced method for total vanadium.					
86. Zinc—Total, milligrams per liter.	Digestion[15] followed by atomic absorption[16] or by colorimetric (Dithizone).	155 ……	148 265	345	159	[3](619) [10](37)
87. Zinc—Dissolved, milligrams per liter.	0.45 micron filtration[17] followed by referenced method for total zinc.					
88. Nitrate (as N), milligrams per liter.	Cadmium reduction; brucine sulfate; automated cadmium or hydrazine reduction.[21]	201 197 207	423 427 620	 338	 119	 [3](624) [10](28)
89. Nitrite (as N), milligrams per liter.	Manual or automated colorimetric (Diazotization).	215	434		121	
90. Oil and grease, milligrams per liter.	Liquid-liquid extraction with trichloro-trifluoroethane-gravimetric.	229	515			
91. Organic carbon; total (TOC), milligrams per liter.	Combustion—Infrared method.[23]	236	532	467	[22] (4)	
92. Organic nitrogen (as N), milligrams per liter.	Kjeldahl nitrogen minus ammonia nitrogen.	175, 159	437		122	[3] (612, 614)
93. Orthophosphate (as P), milligrams per liter.	Manual or automated ascorbic acid reduction.	249 256	451 624	354	131	[3] (621)
94. Pentachlorophenol, milligrams per liter.	Gas chromatography[11]					
95. Pesticides, milligrams per liter.	do.[11]		555	529	[22] (21)	
96. Phenols, milligrams per liter	Distillation followed by Colorimetric. (4AAP)	241	574	545		
97. Phosphorus (elemental), milligrams per liter.	Gas chromatography[24]					
98. Phosphorus; total (as P), milligrams per liter.	Persulfate digestion followed by manual or automated ascorbic acid reduction.	249 256	476, 451 624	354	133	[3] (621)
RADIOLOGICAL						
99. Alpha—Total, pCi per liter	Proportional or scintillation counter.		648	591	[11][12] (75+78)	
100. Alpha—Counting error, pCi per liter.	do.		648	594	[11] (79)	
101. Beta—Total, pCi per liter	Proportional counter		648	601	[11][12] (75+78)	
102. Beta—Counting error, pCi per liter.	do.		648	606	[11] (79)	
103. (a) Radium—Total, pCi per liter.	do.		661	661		
(b) ^{226}Ra, pCi per liter	Scintillation counter		667		[11] (81)	

See footnotes at end of table.

TABLE I.—*List of approved test procedures*[1]—Continued

Parameter and units	Method	1974 EPA methods	11th ed. standard methods	References (page nos.)		Other approved methods[3]
				Pt. 31 1975 ASTM	USGS methods[2]	
RESIDUE						
104. Total, milligrams per liter	Gravimetric, 103 to 105° C.	270	91			
105. Total dissolved (filterable), milligrams per liter.	Glass fiber filtration, 180° C.	266	92			
106. Total suspended (nonfilterable), milligrams per liter.	Glass fiber filtration, 103 to 105° C.	268	94			
107. Settleable, milliliters per liter or milligrams per liter.	Volumetric or gravimetric		95			
108. Total volatile, milligrams per liter.	Gravimetric, 550° C.	272	95			
109. Specific conductance, micromhos per centimeter at 25° C.	Wheatstone bridge conductimetry.	275	71	120	148	[3] (606)
110. Sulfate (as SO_4), milligrams per liter.	Gravimetric; turbidimetric;		493	424		[3] (624)
	or automated colorimetric	277	496	425		[3] (625)
	(barium chloranilate).	279				
111. Sulfide (as S), milligrams per liter.	Titrimetric—iodine for levels greater than 1 mg per liter;	284	505		154	
	Methylene blue photometric.		503			
112. Sulfite (as SO_3), milligrams per liter.	Titrimetric, iodine-iodate	285	508	435		
113. Surfactants, milligrams per liter.	Colorimetric (Methylene blue).	157	600	494	[11] (11)	
114. Temperature, degrees C	Calibrated glass or electrometric thermometer.	286	125		[11] (31)	
115. Turbidity, NTU	Nephelometric	295	132	223	156	

[1] Recommendations for sampling and preservation of samples according to parameter measured may be found in "Methods for Chemical Analysis of Water and Wastes, 1974" U.S. Environmental Protection Agency, table 2, pp. viii-xii.

[2] All page references for USGS methods, unless otherwise noted, are to Brown, E., Skougstad, M. W., and Fishman, M. J., "Methods for Collection and Analysis of Water Samples for Dissolved Minerals and Gases," U.S. Geological Survey Techniques of Water-Resources Inv., book 5, ch. A1, (1970).

[3] EPA comparable method may be found on indicated page of "Official Methods of Analysis of the Association of Official Analytical Chemists" methods manual, 12th ed. (1975).

[4] Manual distillation is not required if comparability data on representative effluent samples are on company file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies.

[5] The method used must be specified.

[6] The 5 tube MPN is used.

[7] Slack, K. V. and others, "Methods for Collection and Analysis of Aquatic Biological and Mircobiological Samples: U.S. Geological Survey Techniques of Water-Resources Inv. book 5, ch. A4 (1973)."

[8] Since the membrane filter technique usually yields low and variable recovery from chlorinated wastewaters, the MPN method will be required to resolve any controversies.

[9] Adequately tested methods for benzidine are not available. Until approved methods are available, the following interim method can be used for the estimation of benzidine: (1) "Method for Benzidine and Its Salts in Wastewaters," available from Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

[10] American National Standard on Photographic Processing Effluents, Apr. 2, 1975. Available from ANSI, 1430 Broadway, New York, N.Y. 10018.

[11] Fishman, M. J. and Brown, Eugene, "Selected Methods of the U.S. Geological Survey for Analysis of Wastewaters," (1976) open-file report 76–177.

[12] Procedures for pentachlorophenol, chlorinated organic compounds, and pesticides can be obtained from the Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

[13] Color method (ADMI procedure) available from Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

[14] For samples suspected of having thiocyanate interference, magnesium chloride is used as the digestion catalyst. In the approved test procedure for cyanides, the recommended catalysts are replaced with 20 ml of a solution of 510 g/l magnesium chloride ($MgCl_2 \cdot 6H_2O$). This substitution will eliminate thiocyanate interference for both total cyanide and cyanide amenable to chlorination measurements.

[15] For the determination of total metals the sample is not filtered before processing. Because vigorous digestion procedures may result in a loss of certain metals through precipitation, a less vigorous treatment is recommended as given on p. 83 (4.1.4) of "Methods for Chemical Analysis of Water and Wastes" (1974). In those instances where a more vigorous digestion is desired the procedure on p. 82 (4.1.3) should be followed. For the measurement of the noble metal series (gold, iridium, osmium, palladium, platinum, rhodium and ruthenium), an aqua regia digestion is to be substituted as follows: Transfer a representitive aliquot of the well-mixed sample to a Griffin beaker and add 3 ml of concentrated redistilled HNO_3. Place the beaker on a steam bath and evaporate to dryness. Cool the beaker and cautiously add a 5 ml portion of aqua regia. (Aqua regia is prepared immediately before use by carefully adding 3 volumes of concentrated HCl to one volume of concentrated HNO_3.) Cover the beaker with a watch glass and return to the steam bath. Continue heating the covered beaker for 50 min. Remove cover and evaporate to dryness. Cool and take up the residue in a small quantity of 1:1 HCl. Wash down the beaker walls and watch glass with distilled water and filter the sample to remove silicates and other insoluble material that could clog the atomizer. Adjust the volume to some predetermined value based on the expected metal concentration. The sample is now ready for analysis.

[16] As the various furnace devices (flameless AA) are essentially atomic absorption techniques, they are considered to be approved test methods. Methods of standard addition are to be followed as noted in p. 78 of "Methods for Chemical Analysis of Water and Wastes," 1974.

[17] Dissolved metals are defined as those constituents which will pass through a 0.45 μm membrane filter. A prefiltration is permissible to free the sample from larger suspended solids. Filter the sample as soon as practical after collection using the first 50 to 100 ml to rinse the filter flask. (Glass or plastic filtering apparatus are recommended to avoid possible contamination.) Discard the portion used to rinse the flask and collect the required volume of filtrate. Acidify the filtrate with 1:1 redistilled HNO_3 to a pH of 2. Normally, 3 ml of (1:1) acid per liter should be sufficient to preserve the samples.

[18] See "Atomic Absorption Newsletter," vol. 13, 75 (1974). Available from Perkin-Elmer Corp., Main Ave., Norwalk, Conn. 06852.

[19] Method available from Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

[20] Recommended methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/l and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to a pH of 12. Therefore, for levels of silver above 1 mg/l 20 ml of sample should be diluted to 100 ml by adding 40 ml each of 2M $Na_2S_2O_3$ and 2M NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/l the recommended method is satisfactory.

[21] An automated hydrazine reduction method is available from the Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

[22] A number of such systems manufactured by various companies are considered to be comparable in their performance. In addition, another technique, based on combustion-methane detection is also acceptable.

[23] Goerlitz, D., Brown, E., "Methods for Analysis of Organic Substances in Water": U.S. Geological Survey Techniques of Water-Resources Inv., book 5, ch. A3 (1972).

[24] R. F. Addison and R. G. Ackman, "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography," "Journal of Chromatography," vol. 47, No. 3, pp. 421-426, 1970.

[25] The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only suspended. Therefore, the 2 results must be added together to obtain "total."

[26] Stevens, H. H., Ficke, J. F., and Smoot, G. F., "Water Temperature—Influential Factors, Field Measurement and Data Presentation: U.S. Geological Survey Techniques of Water Resources Inv., book 1 (1975)."

[38 FR 28758, Oct. 16, 1973, as amended at 41 FR 52781, Dec. 1, 1976; 42 FR 3306, Jan. 18, 1977]

§ 136.4 Application for alternate test procedures.

(a) Any person may apply to the Regional Administrator in the Region where the discharge occurs for approval of an alternative test procedure.

(b) When the discharge for which an alternative test procedure is proposed occurs within a State having a permit program approved pursuant to section 402 of the Act, the applicant shall submit his application to the Regional Administrator through the Director of the State agency having responsibility for issuance of NPDES permits within such State.

(c) Unless and until printed application forms are made available, an application for an alternate test procedure may be made by letter in triplicate. Any application for an alternate test procedure under this paragraph (c) shall:

(1) Provide the name and address of the responsible person or firm making the discharge (if not the applicant) and the applicable ID number of the existing or pending permit, issuing agency, and type of permit for which the alternate test procedure is requested, and the discharge serial number.

(2) Identify the pollutant or parameter for which approval of an alternate testing procedure is being requested.

(3) Provide justification for using testing procedures other than those specified in Table I.

(4) Provide a detailed description of the proposed alternate test procedure, together with references to published studies of the applicability of the alternate test procedure to the effluents in question.

(d) An application for approval of an alternate test procedure for nationwide use may be made by letter in triplicate to the Director, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268. Any application for an alternate test procedure under this paragraph (d) shall:

(1) Provide the name and address of the responsible person or firm making the application.

(2) Identify the pollutant(s) or parameter(s) for which nationwide approval of an alternate testing procedure is being requested.

(3) Provide a detailed description of the proposed alternate procedure, together with references to published or other studies confirming the general applicability of the alternate test procedure to the pollutant(s) or parameter(s) in waste water discharges from representative and specified industrial or other categories.

(4) Provide comparability data for the performance of the proposed alternate test procedure compared to the performance of the approved test procedures.

[38 FR 28760, Oct. 16, 1973, as amended at 41 FR 52785, Dec. 1, 1976]

§ 136.5 Approval of alternate test procedures.

(a) The Regional Administrator of the region in which the discharge will occur has final responsibility for approval of any alternate test procedure proposed by the responsible person or firm making the discharge.

(b) Within thirty days of receipt of an application, the Director will forward such application proposed by the responsible person or firm making the discharge, together with his recommendations, to the Regional Administrator. Where the Director recommends rejection of the application for scientific and technical reasons which he provides, the Regional Administrator shall deny the application, and shall forward a copy of the rejected application and his decision to the Director of the State Permit Program and to the Director of the Environmental Monitoring and Support Laboratory, Cincinnati.

(c) Before approving any application for an alternate test procedure proposed by the responsible person or firm making the discharge, the Regional Administrator shall forward a copy of the application to the Director of the Environmental Monitoring and Support Laboratory, Cincinnati.

(d) Within ninety days of receipt by the Regional Administrator of an application for an alternate test procedure, proposed by the responsible person or firm making the discharge, the Regional Administrator shall notify the applicant and the appropriate State agency of approval or rejection, or shall specify the additional information which is required to determine whether to approve the proposed test procedure. Prior to the expiration of such ninety day period, a recommendation providing the scientific and other technical basis for acceptance or rejection will be forwarded to the Regional Administrator by the Director of the Environmental Monitoring and Support Laboratory, Cincinnati. A copy of all approval and rejection notifications will be forwarded to the Director, Environmental Monitoring and Support Laboratory, Cincinnati, for the purposes of national coordination.

(e) Within ninety days of the receipt by the Director of the Environmental Monitoring and Support Laboratory, Cincinnati of an application for an alternate test procedure for nationwide use, the Director of the Environmental Monitoring and Support Laboratory, Cincinnati shall notify the applicant of his recommendation to the Administrator to approve or reject the application, or shall specify additional information which is required to determine whether to approve the proposed test procedure. After such notification, an alternate method determined by the Administrator to satisfy the applicable requirements of this part shall be approved for nationwide use to satisfy the requirements of this subchapter; alternate test procedures determined by the Administrator not to meet the applicable requirements of this part shall be rejected. Notice of these determinations shall be submitted for publication in the FEDERAL REGISTER not later than 15 days after such notification and determination is made.

[38 FR 28760, Oct. 16, 1973, as amended at 41 FR 52785, Dec. 1, 1976]

Section E. NPDES Sampling and Analytical Methods

I. *Sampling.* EPA has not yet promulgated many sampling methodologies under section 304(h) of the CWA. This section gives some general guidance on good sampling techniques; contact your EPA or State permitting authority for more detailed guidance or for answers to specific questions. Where the approved methods referenced in Table 5 include requirements on how samples should be collected and preserved, follow the requirements whenever you use those methods.

A. *General considerations.* 1. Choose a time for sampling that is representative of your normal operation. All operations and processes which contribute wastewater should be in normal operation, to the extent feasible. Also, your treatment system should be operating properly with no system upsets.

2. The sample should be collected where the wastewater is well mixed: near the center of the flow channel, where turbulence is at a maximum.

3. The location for sampling should be adequate for the collection of a representative sample; the site specified in the permit if there is one.

4. Sampling for ponds and lagoons should be by composite samples unless past studies have proved that the retention time is long enough that the discharge is homogeneous.

5. The volume of the collected sample should be sufficient to perform all required analyses, including quality control and split sampling. Consult the laboratory receiving the sample and the methods in Table 5 for specific volume requirements.

6. Sample containers must be made of appropriate materials and cleaned thoroughly to prevent any contamination or change in volume of the sample. See the methods in Table 5 for specific requirements.

7. All samples must be preserved by refrigeration at 4°C from the time of collection (and during collection for composite samples) until the time of analysis.

B. *Types of Samples.* 1. Grab—An individual sample collected at a randomly-selected time over a period of time not exceeding 15 minutes. The sample volume must be at least 100 ml, and adequate for all required analyses. Grab samples are analyzed separately, and the average is computed with flow-weighted values.

2. Grab composite—A series of grab samples collected over the sampling period, and composited prior to analysis. For volatile organic toxics, follow the compositing method in the

3. 24-hour composite—A composite of samples collected at periodic intervals, over a 24-hour period. The composite must be flow proportional, with either the time interval between aliquots or the volume of aliquots proportional to the stream flow at the time of sampling, or to the total stream flow since the last aliquot. The number of aliquots shall be no less than eight, and in all cases as specified by the methods in Table 5. The minimum volume of each aliquot is 100 ml. The sample collection may be done either by manually collecting the periodic aliquots or by using an automatic sampler. More than eight aliquots will be required when the wastewater loading is highly variable.

For periods of flow less than 24 hours, collect flow proportional aliquots over the entire period of flow, with no less than eight aliquots.

4. 72-hour composite—May be collected in the following three ways:

a. A continuous composite, wth the same requirements as specified in (3) above for 24-hour composites, except that there shall be no less than 24 aliquots collected in the 72-hour period; or

b. Three consecutive 24-hour composites collected as specified in (3) above, combined prior to analysis in either equal volumes or flow proportional volumes; or

c. For non-volatile organic toxic pollutants, three consecutive 24-hour composites collected as specified in (3) above, each extracted with methylene chloride according to the methods in Table 5 at the conclusion of each 24-hour compositing period, with the three extracts combined for further sample preparation and analysis.

II. *Analytical Methods.* The following table represents the analytical methods promulgated under 40 CFR Part 136 as of May 1979. Revisions and additions to the methods will be proposed starting in Summer 1979 (see footnote 14), and when these changes are final, they will be incorporated into the table.

The following table has been included in these instructions for the convenience of permit applications, but it does not consititute the official listing of approved methods. Refer to 40 CFR Part 136 for the complete listing, which includes a brief description of each method, as well as some further details of analytical procedures in footnotes.

Alternate test procedures to those listed here and in 40 CFR Part 136 may be approved for use on an individual basis or for nationwide use. Refer to 40 CFR Part 136 for prodedures and requirements concerning application for approval of alternate test procedures.

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SAMPLING AND ANALYTICAL METHODS

Parameter (units)	Sample type	Analytical Methods—References									
		1974 EPA Methods(1)*		14th ed., Standard Methods(2)		Pt. 31, 1975 ASTM(3)		USGS Methods(4)		Other Approved Methods	
		Page Number	Detection Limit	Page Number	Detection Limit	Page Number	Detection Limit	Page Number	Detection Limit	Page Number	Detection Limit
Biochemical Oxygen Demand (BOD_5) (mg/l)	24 hour composite	-	-	543	-	-	-	-	-	17(6)	-
Chemical Oxygen Demand (COD) (mg/l)	24 hour composite	20	5 mg/l	550	10 mg/l	472	-	124	-	610(7) 17(6)	5 mg/l -
pH (Standard Units)	Grab	239	-	460	-	178	-	129	-	606(7)	-
Total Suspended Solids (TSS) (mg/l)	24 hour composite	268	10 mg/l	94	-	-	-	-	-	-	-
Temperature (°C)	Grab	286	-	125	-	-	-	31(8)	-	-	-
Total Kjeldahl Nitrogen (as N) (TKN) (mg/l)	24 hour composite	165 175 182	0.03 mg/l 1 mg/l 0.05 mg/l	437	0.02 mg/l	-	-	122	-	612(7)	-
Total Organic Carbon (TOC) (mg/l)	24 hour composite	236	1 mg/l	532	1 mg/l	467	2 mg/l	4(9)	1 mg/l	-	-
Cyanide, Total (mg/l)	Grab	40 40	1 mg/l 0.02 mg/l	361 361	1 mg/l 0.02 mg/l	503 503	1 mg/l -	85	0.01 mg/l	22(6)	-
Phenols, Total (4AAP) (mg/l)	Grab	241	0.005 mg/l	582	0.007 mg/l	545	0.005 mg/l	-	-	-	-

*All footnotes are at the end of the table.

Parameter (units)	Sample type	Analytical Methods—References									
		1974 EPA Methods(1)		14th ed., Standard Methods(2)		Pt. 31, 1975 ASTM(3)		USGS Methods(4)		Other Approved Methods	
		Page Number	Detection Limit	Page Number	Detection Limit	Page Number	Detection Limit	Page Number	Detection Limit	Page Number	Detection Limit
Volatile toxic pollutants(13,14)	72 hour grab composite	-	-	-	-	-	-	-	-	-	-
Acid, base-neutral, pesticides organic toxic pollutants (13,14)	72 hour composite or 3 24-hour composites	-	-	-	-	-	-	-	-	-	-
Antimony, Total (mg/l)	24 hour composite	94	0.5 mg/l	-	-	-	-	-	-	-	-
Arsenic, Total (mg/l)	24 hour composite	9 95	0.01 mg/l 0.002 mg/l	283 159	0.03 mg/l 0.0025 mg/l	-	-	31(10) 37(10)	0.005mg/l 0.001mg/l	-	-
Beryllium, Total (mg/l)	24 hour composite	99	0.025 mg/l	152 177	0.03 mg/l 0.005 mg/l	-	-	53	0.01 mg/l	-	-
Cadmium, Total (mg/l)	24 hour composite	101	0.025 mg/l	148 182	0.025 mg/l 0.01 mg/l	345	0.05 mg/l	62	0.025 mg/l	619(7) 37(6)	0.1 mg/l -
Chromium, Total (mg/l)	24 hour composite	105	0.1 mg/l	148 192	0.1 mg/l -	345 286	0.2 mg/l -	78 77	-	619(7)	1 mg/l
Copper, Total (mg/l)	24 hour composite	108	0.1 mg/l	148 196	0.1 mg/l 0.03 mg/l	345 293	0.1 mg/l 0.05 mg/l	83	0.05 mg/l	619(7) 37(6)	0.1 mg/l -
Lead, Total (mg/l)	24 hour composite	112	0.5 mg/l	148 215	0.5 mg/l -	345	0.34 mg/l	105	0.001 mg/l	619(7)	1 mg/l
Mercury, Total (mg/l)	24 hour composite	118	0.0002 mg/l	156	-	338	0.0002 mg/l	51(10)	0.0001 mg/l	-	-
Nickel, Total (mg/l)	24 hour composite	141	0.15 mg/l	148	0.1 mg/l	345	0.08 mg/l	115	0.001 mg/l	-	-

arameter (units)	Sample type	Analytical Methods—References									
		1974 EPA Methods(1)		14th ed., Standard Methods(2)		Pt. 31, 1975 ASTM(3)		USGS Methods(4)		Other Approved Methods	
		Page Number	Detection Limit	Page Number	Detection Limit	Page Number	Detection Limit	Page Number	Detection Limit	Page Number	Detection Limit
Selenium, Total (mg/l)	24 hour composite	145	0.002 mg/l	159	0.0025 mg/l	-	-	-	-	-	-
Silver, Total (mg/l)	24 hour composite	146	0.06 mg/l	148 243	0.06 mg/l -	-	-	142	0.001 mg/l	619(7) 37(6)	0.1 mg/l -
Thallium, Total (mg/l)	24 hour composite	149	0.05 mg/l	-	-	-	-	-	-	-	-
Zinc, Total (mg/l)	24 hour composite	155	0.02 mg/l	148 265	0.015 mg/l -	345	0.02 mg/l	159	0.01 mg/l	619(7) 37(6)	0.1 mg/l -
Ammonia (as N) (mg/l)	24 hour composite	158 159 165 168	0.05 mg/l 1 mg/l 0.03 mg/l 0.01 mg/l	410 412 416	- 0.02 mg/l 0.01 mg/l	237	-	116	-	614(7)	0.05 mg/l
Asbestos(14) (fibers/l)	24 hour composite	-	-	-	-	-	-	-	-	-	-
Bromide (mg/l)	24 hour composite	14	2 mg/l	-	-	323	50 mg/l	58	1 mg/l	-	-
Chlorine, Total Residual (mg/l)	Grab	35 55	- -	318 322 332 329	1 mg/l - - -	278	-	-	-	-	-
Color (platinum cobalt units)	24 hour composite	39 36	- 1 unit	66 64	- 1 unit	-	-	82	-	-	-

Parameter (units)	Sample type	Analytical Methods—References									
		1974 EPA Methods(1)		14th ed., Standard Methods(2)		Pt. 31, 1975 ASTM(3)		USGS Methods(4)		Other Approved Methods	
		Page Number	Detection Limit	Page Number	Detection Limit	Page Number	Detection Limit	Page Number	Detection Limit	Page Number	Detection Limit
Fecal Coliform (number per 100 ml)	Grab	-	-	922 937	2.2 per 100 ml -	-	-	45(5)	1 per 100 mg	-	-
Fluoride mg/l	24 hour composite	65 59 61	0.1 mg/l 0.1 mg/l 0.05 mg/l	389 391 393 614	- 0.2 mg/l 0.05 mg/l 0.1 mg/l	307 305	0.1 mg/l -	93	0.1 mg/l	-	-
Nitrate (as N) (mg/l)	24 hour composite	201 197 207	0.01 mg/l 0.1 mg/l 0.05 mg/l	427 620 423	0.1 mg/l 0.5 mg/l 0.002 mg/12	358	1 mg/l	119	-	614(7) 28(6)	0.1 mg/l 1 mg/l
Nitrite (as N) (mg/l)	24 hour composite	215	0.01 mg/l	434	0.001 mg/l	-	-	121	-	-	-
Oil and Grease (mg/l)	Grab or Grab composite	229	5 mg/l	515	-	-	-	-	-	-	-
Phosphorus, Total (as P) (mg/l)	24 hour composite	249 256	0.01 mg/l 0.001 mg/l	476 481 624	- 0.01 mg/l 0.001 mg/l	384	0.01 mg/l	130	-	621(7)	0.01 mg/l
Radioactivity Alpha-Total (pCi/l)	24 hour composite	-	-	648	-	591	500 pCi/l	75, 78 (10,11)	-	-	-
Alpha-Counting Error (pCi/l)	24 hour composite	-	-	648	-	594	-	79(10)	-	-	-
Beta-Total (pCi/l)	24 hour composite	-	-	648	-	601	500 pCi/l	75, 78 (10,11)	-	-	-
Beta-Counting Error (pCi/l)	24 hour composite	-	-	648	-	606	-	79(10)	-	-	-
(a) Radium-Total (pCi/l)	24 hour composite	-	-	661	-	661	1 pCi/l	-	-	-	-

Parameter (units)	Sample type	Analytical Methods--References									
		1974 EPA Methods(1)		14th ed., Standard Methods(2)		Pt. 31, 1975 ASTM(3)		USGS Methods(4)		Other Approved Methods	
		Page Number	Detection Limit	Page Number	Detection Limit	Page Number	Detection Limit	Page Number	Detection Limit	Page Number	Detection Limit
(b) ^{226}Ra (pCi/l)	24 hour composite	-	-	667	-	-	-	81(10)		-	-
Sulfate (as SO_4)(mg/l)	24 hour composite	277 279	- 10 mg/l	493 496	10 mg/l 1 mg/l	424 425	20 mg/l 10 mg/l	-	-	624(7) 623(7)	- -
Sulfide (as S) (mg/l)	24 hour composite	284	1 mg/l	505 503	1 mg/l 0.1 mg/l	-	-	154	0.5 mg/l	-	-
Sulfite (as SO_3) (mg/l)	24 hour composite	285	2 mg/l	508	2 mg/l	435	3 mg/l	-	-	-	
Surfactants (mg/l)	24 hour composite	157	0.025 mg/l	600	0.025 mg/l	494	-	11(9)	0.025 mg/l	-	-
Aluminum, Total (mg/l)	24 hour composite	92	1 mg/l	152 171	0.9 mg/l 0.006 mg/l	-	-	19(10)	0.01mg/l	-	-
Barium, Total (mg/l)	24 hour composite	97	0.4 mg/l	152	0.3 mg/l	-	-	52	0.1 mg/l	-	-
Boron, Total (mg/l)	24 hour composite	13	0.1 mg/l	287	0.1 mg/l	-	-	-	-	-	-
Cobalt, Total (mg/l)	24 hour composite	107	0.2 mg/l	148	0.15 mg/l	345	0.25 mg/l	80	-	37(6)	-
Iron, Total (mg/l)	24 hour composite	110	0.12 mg/l	148 208	0.1 mg/l 0.02 mg/l	345 326	0.1 mg/l 0.05 mg/l	102	0.1 mg/l	619(7)	0.1 mg/l
Magnesium, Total (mg/l)	24 hour composite	114	0.007 mg/l	148	0.007 mg/l	345	0.05 mg/l	109	0.05 mg/l	619(7)	0.01 mg/l
Molybdenum, Total (mg/l)	24 hour composite	139	0.3 mg/l	-	-	350	0.001 mg/l	-	-	-	-

Parameter (units)	Sample type	Analytical Methods—References									
		1974 EPA Methods(1)		14th ed., Standard Methods(2)		Pt. 31, 1975 ASTM(3)		USGS Methods(4)		Other Approved Methods	
		Page Number	Detection Limit	Page Number	Detection Limit	Page Number	Detection Limit	Page Number	Detection Limit	Page Number	Detection Limit
Manganese, Total (mg/l)	24 hour composite	116	0.05 mg/l	148 225 227	0.05 mg/l 0.3 mg/l 0.05 mg/l	345	0.04 mg/l	111	0.05 mg/l	619(7)	0.1 mg/l
Tin, Total (mg/l)	24 hour composite	150	4 mg/l	-	-	-	-	65(10)	0.05mg/l	-	-
Titanium, Total (mg/l)	24 hour composite	151	2 mg/l	-	-	-	-	-	-	-	-
Pesticides(12) (ug/l)	24 or 72 hour composite	-	-	555	(13)	529	(13)	30(9)	(13)	-	-
2,3,7,8 Tetrachloro dibenzo-p-dioxin (TCDD)(14) (ug/l)	72 hour composite or 3 24-hour composites	-	-	-	-	-	-	-	-	-	-

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Footnotes

(1) *Methods for Chemical Analysis of Water and Waste*, 1974.

(2) *Standard Methods for the Examination of Water and Waste Water*, 14th Edition, 1976.

(3) *Annual Book of Standards, Part 31, Water*, 1975, by American Society for Testing and Materials.

(4) Unless otherwise noted, Brown, E., Skougstad, M. W., and Fishman, M. J. "Methods for Collection and Analysis of Water Samples for Dissolved Minerals and Gases," U.S. Geological Survey Techniques of Water-Resources Inventory, book 5, Ch. A 1 (1970).

(5) Slack, K. V., and others, "Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples", U.S. Geological Survey Techniques of Water-Resources Inv., book 5, Ch A 4 (1973).

(6) American National Standards on Photographic Processing Effluents, April 2, 1975.

(7) "Official Methods of Analysis of the Association of Official Analytical Chemists" methods manual, 12th ed. (1975).

(8) Stevens, H. H., Ficke, J. F., and Smoot, G. F. "Water Temperature-Influential Factors, Field Measurement and Data Presentation", U.S. Geological Survey Techniques of Water Resources Inventory, book 1 (1975).

(9) Goerlitz, D., Brown, E. "Methods for Analysis of Organic Substances in Water", U.S. Geological Survey Techniques of Water-Resources Inv., book 5, Ch. A 3 (1972).

(10) Fishman, M. J., and Brown, Eugene "Selected Methods of the U.S. Geological Survey for Analysis of Wastewaters" (1976) open-file report 76-177.

(11) The method found on p. 75 measures the dissolved portion and the method on p. 78 measures suspended. Therefore, the two results must be added together to get "total".

(12) Methods available from the Environmental Monitoring and Support Laboratory, the U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

(13) See referenced methods for detection limits of individual pesticides and organic toxic pollutants. In most cases, the 10 μg/l limit in 40 CFR § 122.68(a)(2)(ii)(A) will apply.

(14) Methods for the analysis of the 114 organic toxic pollutants and asbestos are being developed by the Agency's Office of Research and Development. The methods for analysis of organic pollutants will include:

(1) Gas chromatography/mass spectrometry (GC/MS) screening protocol for qualitative analysis for all 114 organic toxics. (An earlier version of this protocol has been used in the screening phase of developing national effluent guidelines.)

(2) Gas chromatography (GC) and liquid chromatography (LC) methods for twelve classes of the organic pollutants, which are being developed by EMSL, Cincinnati and by Effluent Guidelines Division contractors.

(3) Quantitative GC/MS, consisting of quality assurance procedures for use with the GC/MS screening protocol which will make it a quantitative method.

By early Summer 1979 the Agency expects to propose for comment the GC/MS screening protocol and the GC and LC methods developed by EMSL, Cincinnati. Shortly thereafter, the Agency will propose methods for sampling and preservation, and for determining the detection limit of an analytical method, and will update the references used in the existing methods. The other methods described above, as well as a new method for total metals using Inductively Coupled Argon Plasma (ICAP), and a method for asbestos analysis, will be proposed for comment about six months later.

The analysis for organic toxic pollutants required by item V-B of Form 2c will be satisfied by using either the proposed GC/MS screening protocol followed by quantification of those organic toxic pollutants identified as present using the proposed GC or LC methods; or by using the proposed quantitative GC/MS method.

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HANDOUT 10

FORM FOR ASSESSING RELEVANT ANALYTICAL METHODS

POLLUTANT PARAMETER	EPA-APPROVED METHOD	PERMITTEE'S LABORATORY METHOD	IF NOT EPA-APPROVED METHOD, WHY?
1.	1. a.	1.	1.
	b.		
	c.		
	d.		
2.	2. a.	2.	2.
	b.		
	c.		
	d.		
3.	3. a.	3.	3.
	b.		
	c.		
	d.		
4.	4. a.	4	4
	b.		
	c.		
	d.		

HANDOUT 11
(1 of 11)

POTENTIAL PROBLEM AREAS ASSOCIATED WITH POLLUTANT ANALYSIS

Pollutant Parameter Analyzed	Problem Areas
Acidity	• Check calibration of pH meter
Alkalinity (pH 4.5)	• Check that samples are analyzed within a few hours of delivery to lab.
Alkalinity, automated	• Check that samples are refrigerated at 4°C until they are run. Check that samples are filtered through 0.45u membrane before analysis.
Arsenic	• Check to see that industrial waste samples are spiked with a known amount of arsenic to establish adequate recovery.
Boron	• Check to see that samples with total Ca and Mg hardness > 100 mg/l as $CaCo_3$ are passed through cation exchange resin before analysis.
Bromide	• Check to see that samples are stored at 4°C and analyzed as soon as possible.
COD	• Check that flask is cooled during H_2SO_4 addition to reduce loss of volatile organics. • Check that glassware used in test is conditioned by running blanks to eliminate traces of organic materials.
Chloride	• Check that samples suspected of containing sulfites are treated by oxidizing with H_2O_2 to eliminate interference.
Chlorine, total residual	• Check to see that samples are not stored; chlorine determinations must be started immediately after sampling, avoiding light and agitation.

Pollutant Parameter Analyzed	Problem Areas
Color	● Check that samples showing visible turbidity are clarified by centrifugation. ● Check that samples are refrigerated at $4^{\circ}C$.
Cyanide, total	● Check that samples are collected in ≥ 1 liter plastic bottles that have been thoroughly cleansed and rinsed. ● Check that samples have been preserved with 2 ml of 10N NaOH/liter of sample (pH ≥ 12) at time of collection. ● Check that samples are refrigerated at 4°C and analyzed as soon as possible. ● Check that samples are tested for chlorine with KI-starch paper and treated with ascorbic acid if blue color results. ● Check calibration procedure of spectrophotometer for colorimetric measurement.
Dissolved oxygen	● Check that special sampling techniques specified on p. 52 of the "1974 EPA Methods" have been observed and that analyses are completed within 4-8 hours after sampling.
Fluoride, total	● For SPADNS method, need for distillation is up to analyst's judgment. ● Check that addition of highly colored SPADNS reagent is done with utmost accuracy because a small error in reagent addition is the most prominent source of error in this test. ● For automated complexone method, check that 8-hydroxyquinoline addition and subsequent extraction is performed on samples suspected of containing ≥ 0.2 mg aluminum per liter. ● For selective ion electrode method, check calibration of pH meter or selective ion meter having direct concentration scale for fluoride. Also, check that pH 5.0 buffer is added to samples to eliminate interferences due to aluminum, iron, and silicon and other complexes formed at extremes of pH.

Pollutant Parameter Analyzed	Problem Areas
Arsenic (cont'd)	• Check that organic forms of arsenic are converted to inorganic compounds and organic matter is oxidized before beginning the analysis. • Check to see that sample is airtight during evolution of arsine to avoid losses.
Barium	• Check that calibration standards are prepared at the time of analyses using the same type of acid (HCl or HNO_3) and at the same concentration as the samples for analysis. • Check to see that potassium (1000 mg/l) is added to samples and standards alike to control barium ionization in nitrous oxide-acetylene flame. • Check to see that lanthanum (2000 mg/l) is added to samples and standard alike to avoid depression of barium absorbance by phosphate, silicon, and aluminum in acetylene-air flame.
Beryllium	• Check to see that calibration standards are prepared at the time of analysis using the same type of acid (HCl or HNO_3) and at the same concentration as the samples for analysis. • Check to see that samples are acidified to pH 1.5 to eliminate interference of bicarbonate ion.
Cadmium	• Check to see that calibration standards are prepared at the time of analysis using the same type of acid (HCl or HNO_3) and at the same concentration as the samples for analysis. • Check to see that extraction procedure is used for cadmium levels < 20 ug/l.
Calcium	• Check that both standards and samples are prepared in dilute HCl solution. • Check that phosphate, sulfate, and aluminum interferences are masked by addition of lanthanum. • Check that ionization interferences are controlled by adding a large amount of alkali to both samples and standards.

Pollutant Parameter Analyzed	Problem Areas
Hardness, total	• Check that routine addition of sodium cyanide solution is done to prevent potential interferences from heavy metals.
METALS	• Check calibration of atomic absorption spectrophotometer. • For determination of dissolved metals, check that samples are filtered through a 0.45 u membrane filter as soon as practical after collection and before analysis. • For determination of suspended metals, check that a representative volume of unpreserved sample is filtered through a 0.45 u membrane filter and the insoluble material is digested with HNO_3 and HCl and diluted prior to analysis.
Aluminum	• Check that calibration standards are prepared at the time of analysis using the same type of acid (HCl or HNO_3) and at the same concentration as the samples for analysis. • Check that 1 mg potassium/ml solution is added to both sample and standard solutions to control ionization of aluminum in the nitrous oxide acetylene flame.
Antimony	• Check that calibration standards are prepared at the time of analysis using the same type of acid (HCl or HNO_3) and at the same concentration as the samples for analysis. • For analysis of samples that contain lead (1000 mg/l) check that 231.1 nm antimony line is used. • Check that acid concentration in samples and standards is matched.
Arsenic	• Check that industrial waste samples are spiked with a known amount of arsenic to establish adequate recovery.

Pollutant Parameter Analyzed	Problem Areas
Chromium	• Check that calibration standards are prepared at the time of analysis using the same type of acid (HCl or HNO_3) and at the same concentration as the samples for analysis. • The nitrous oxide-acetylene flame provides the greatest freedom from chemical and matrix interferences. • Check that 1% ammonium bifluoride in 0.2% sodium sulfate is added to samples to control the suppression of both Cr (III) and Cr (VI) absorption by most interfering ions when a fuel rich air-acetylene flame is used. • Check that the extraction procedure is used for chromium levels < 50 mg/l and that an oxidation step is included to measure trivalent chromium.
Cobalt	• Check that calibration standards are prepared at the time of analysis using the same type of acid (HCl or HNO_3) and at the same concentration as the samples for analysis. • Check that the extraction procedure is used for cobalt levels < 50 mg/l.
Copper	• Check that calibration standards are prepared at the time of analysis using the same type of acid (HCl or HNO_3) and at the same concentration as the samples for analysis. • Check that the extraction procedure is used for copper levels < 20 mg/l.
Iron	• Check that calibration standards are prepared at the time of analysis using the same type of acid (HCl or HNO_3) and at the same concentration as the samples for analysis. • Better signal-to-noise ratio can be obtained from a neon-filled hollow cathode lamp than an argon filled lamp.
Lead	• Check that calibration standards are prepared at the time of analysis using the same type of acid (HCl or HNO_3) and at the same concentration as the samples for analysis. • Check that care is taken to position the light beam in the most stable, center portion of the flame because analysis for lead is very sensitive to turbulence and absorption bands in the flame. The burner should first be adjusted to maximize the absorbance reading

Pollutant Parameter Analyzed	Problem Areas
	with a lead standard; then a water blank should be aspirated and fine adjustments made in the burner alignment to minimize the signal. • Check to see that extraction at pH 2.8 is used for lead levels <100 ug/l.
Magnesium	• Note that analytical sensitivity decreases with increased lamp current. • Check that interferences caused by aluminum concentrations > 2 mg/l are masked by addition of lanthanum.
Manganese	• Check that calibration standards are prepared at the time of analysis using the same type of acid (HCl or HNO_3) and at the same concentration as the samples for analysis. • Check that extraction at pH 4.5-5 is carried out for levels of manganese < 25 mg/l and analysis is made without delay to prevent resolution of manganese chelate in aqueous phase.
Mercury, manual cold vapor technique	• Check that organomercury compounds are oxidized to mercuric ion by potassium persulfate before measurement. • Check that the samples are preserved by acidification with nitric acid to pH ≤ 2 immediately upon collection. Check that dissolved mercury samples are filtered through an all glass apparatus before the acid is added. • Check that possible interference from sulfide has been eliminated by the addition of potassium permanganate. • Check that ∿25 ml excess permanganate solution has been added to sea waters, brines, and industrial effluents high in chlorides. • Check that care is taken to assure that free chlorine is absent before mercury reduction; this should be accomplished by using an excess of hydroxylamine sulfate reagent (∿25 ml). • Check to make certain that dead air space in the BOD bottle has been purged before addition of stannuous sulfate.

Pollutant Parameter Analyzed	Problem Areas
	• Check to see that a preliminary run without reagents is made to identify possible interference from certain volatile organic materials. If an interference is found, the sample should be analyzed both by using the regular procedure and again under oxidizing conditions only (without the reducing agents). The true mercury value is obtained by subtracting the two values. • Check to see that laboratory follows directions for disposal of mercury-containing wastes given in ASTM Standards, Part 23, Water and Atmospheric Analysis, p. 352, Method D3223 (1973).
Mercury, automated cold vapor technique	See above, Mercury, manual cold vapor technique plus following: • Check to ensure that formation of a heavy precipitate does not occur upon addition of concentrated sulfuric acid to the sample; if such a precipitate is encountered, the problem sample cannot be analyzed by the automated method. • Check to see that samples containing solids are blended and then mixed while being sampled if total mercury values are to be reported.
Molybdenum	• Check to see that calibration standards are prepared at the time of analysis using the same type of acid (HCl or HNO_3) and at the same concentration as the samples for analysis. • Check to see that with a nitrous oxide-acetylene flame, interferences of calcium and other ions are controlled by adding 1000 mg aluminum/liter of solution.
Nickel	• Check to see that calibration standards are prepared at the time of analysis using the same type of acid (HCl or HNO_3) and at the same concentration as the samples for analysis. • Check to see that the extraction procedure is used for levels of nickel below 50 ug/l.
Potassium	• Check that samples are preserved at pH 2 with 1:1 nitric acid at the time of collection. • Check that dilutions of stock solution to be used as calibration standards are prepared at the time of analysis. • Check that any high-level sodium interference is compensated by approximately matching the

Pollutant Parameter Analyzed	Problem Areas
	sodium content of the potassium standards with that of the sample. • Check that possible enhancement effects of Na, Li, and Cs, especially in a high temperature fuel rich flame, are eliminated by setting the burner assembly approximately 0.05 cm below the optical light path so that the optical light path is sliced at the bottom by the burner head. To cover the range of potassium values normally observed in surface waters (0.1 - 20 mg/l), it is suggested that the burner be rotated 75°.
Selenium	• Check that organic forms of selenium are converted to an inorganic form and organic matter is oxidized before beginning the analysis.
Silver	• Check that dilutions of the stock solution to be used as calibration standards are prepared at the time of analysis and maintained at an acid strength of 0.15% HNO_3. Diluted solutions are not stable and should be discarded after use. • Check that extraction procedure is used for levels of silver < 20 ug/l.
Sodium	• Check that samples are preserved at pH 2 with 1:1 nitric acid at the time of collection. • Check that dilutions of stock solution to be used as calibration standards are prepared at the time of analysis. • Note that low temperature flames increase sodium sensitivity by reducing ionization. If a high temperature flame is used, check that ionization is controlled by adding potassium (1000 mg/l) to both standards and samples.
Thallium	• Check that calibration standards are prepared at the time of analysis using the same type of acid (HCl or HNO_3) and at the same concentration as the samples for analysis.
Tin	• Check that dilutions of stock tin solution to be used as calibration standards are prepared at the time of analysis and maintained at an acid concentration of 10% HCl.
Titanium	• Check that dilutions of stock titanium solution to be used as calibration standards are prepared at the time of analysis.

Pollutant Parameter Analyzed	Problem Areas
	• Check that potassium (1000 mg/l) is added to standards and samples alike to control enhancement effects of many interfering elements.
Vanadium	• Check that calibration standards are prepared at the time of analysis using the same type of acid (HCl or HNO_3) and at the same concentration as the samples for analysis. • Check that enhancement effects of aluminum and titanium interferences are controlled by adding excess aluminum (1000ppm) to both samples and standards.
Zinc	• Check that calibration standards are prepared at the time of analysis using the same type of acid (HCl or HNO_3) and at the same concentration as the samples for analysis. • Note that zinc sensitivity may be increased by the use of low-temperature flames.
Ammonia as Nitrogen, distillation procedure	• Check to see that samples are preserved with H_2SO_4 or $HgCl_2$ and refrigerated at 4°C before analysis. • Note that direct nesslerization without distillation invalidates the method due to the fact that many aromatic, alphatic, and other compounds, both organic and inorganic, will cause turbidity upon the addition of Nessler reagent. • Check to see that interfering volatile alkaline compounds such as certain ketones, adlehydes, and alcohols are eliminated by boiling off at pH 2-3 prior to distillation and nesslerization. • Check to see that residual chlorine has been removed by pretreatment of the sample with sodium thiosulfate before distillation. • If sample has been preserved with a mercury salt, check to see that the mercury ion is complexed with sodium thiosulfate prior to distillation. • Check calibration of spectrophotometer or filter photometer.
Ammonia as Nitrogen, selective ion electrode method	• Check that samples are refrigerated at 4°C and analyzed within 24 hours or preserved with 2 ml concentrated H_2SO_4 per liter (pH <2) for a longer preanalysis period.

Pollutant Parameters Analyzed	Problem Areas
	• Note that samples must not be preserved with mercuric chloride because mercury interferes by forming a strong complex with ammonia. • Check calibration of electrometer.
Ammonia as Nitrogen, automated colorimetric phenolate method	• Check that samples are preserved by addition of 2 ml concentrated H_2SO_4 or 40 mg $HgCl_2$ per liter and refrigeration at 4°C. • Check to see that interference from $HgCl_2$ preservative is overcome by adding a comparable amount of $HgCl_2$ to the ammonia standards used for the preparation of the standard curve. • Check that calcium and magnesium precipitation problems are eliminated for sea water samples by using 5% EDTA. • Check that sample turbidity is removed by filtration prior to analysis. • Check to ensure that distilled water is ammonia-free. Distilled water should be passed through an ion exchange column comprised of a mixture of both strongly acidic cation and strongly basic anion exchange resins.
Kjeldahl Nitrogen, total	• Check that samples have been preserved by addition of 2 ml of concentrated H_2SO_4 or 40 mg $HgCl_2$ per liter and stored at 4°C. Preserved samples should be analyzed as soon as possible to avoid conversion of organic nitrogen to ammonia. • Check to ensure that distilled water is ammonia-free. Distilled water should be passed through an ion exchange column comprised of a mixture of both strongly acidic cation and strongly basic anion exchange resins. • Check that distillation apparatus is pre-steamed before use by distilling a 1:1 mixture of distilled water and sodium hydroxide-sodium thiosulfate solution until the distillate is ammonia-free. This operation should be repeated each time the apparatus is out of service long enough to accumulate ammonia (usually 4 hours or more).

Pollutant Parameter Analyzed	Problem Areas
Kjeldahl Nitrogen, total, automated phenate method	• Check that samples are preserved by addition of 2 ml of concentrated H_2SO_4 or 40 mg $HgCl_2$ per liter and refrigeration at 4°C. • Check to ensure that distilled water is ammonia-free. Distilled water should be passed through and ion exchange column comprised of a mixture of both strongly acidic cation and strongly basic anion exchange resins. Dowex XE-75 or equivalent should also be used to remove organic impurities.

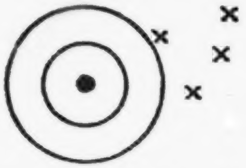
HANDOUT 12

DEFINITIONS

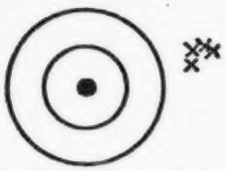
ACCURACY - PRECISION - ERROR

Accuracy - degree of agreement between measured value and true value

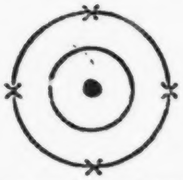
Precision - degree of agreement among results obtained by repeated measurements on a single sample under a given set of conditions



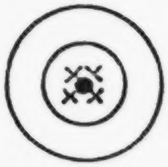
IMPRECISE AND INACCURATE



PRECISE BUT INACCURATE



ACCURATE BUT IMPRECISE



PRECISE AND ACCURATE

Figure 1. PRECISION AND ACCURACY

HANDOUT 13

EXAMPLE OF BENCH SHEET

NL-C-88
(7-68)
Spectrographic Analyses Bench Data

Sample #_____Date_____________Source____________________TDS________

___________ml. conc. to______________ml. Factor__________________

	Test Count	Sec
1.		
2.		
3.		

	Count			Rerun Count			Av. Count	PPM In Conc. Sample	Less Than	(µg/l)PPB Orig. Sample
1. Zn									☐	☐☐☐.☐
2. Cd									☐	☐☐☐.☐
3. As									☐	☐☐☐.☐
4. B									☐	☐☐☐.☐
5. P									☐	☐☐☐.☐
6. Fe									☐	☐☐☐.☐
7. Mo									☐	☐☐☐.☐
8. Mn									☐	☐☐☐.☐
9. Al									☐	☐☐☐.☐
10. Be									☐	☐☐.☐☐
11. Cu									☐	☐☐☐.☐
12. Ag									☐	☐☐☐.☐
13. Ni									☐	☐☐☐.☐
14. Co									☐	☐☐☐.☐
15. Pb									☐	☐☐☐.☐
16. Cr									☐	☐☐☐.☐
17. V									☐	☐☐☐.☐
18. Ba									☐	☐☐☐.☐
19. Sr									☐	☐☐☐.☐

LABORATORY SERVICES

Distilled Water

- In metal stills, surfaces in contact with the distillate should be coated with tin to prevent contamination

- Glass stills are preferable to metal but have limited capacity

- All stills require periodic cleaning

- Pretreating the incoming feed water will improve the quality of distilled water. For example, carbon filters will remove organic materials. A mined-bed ion exchanger will remove ions.

- If the distilled water is piped into the laboratory it should not be contaminated by the pipes. The pipes should be tin lines or chemically resistant glass.

- Where the distilled water is stored in the laboratory borosilicate-free glass containers should be used to prevent contamination. These containers should be covered and have a filter in the air vent to remove airborne dust, gases and fumes.

- Distilled water for certain analysis is required to be ammonia-free, carbon dioxide-free or ion-free. The procedures for making this distilled water are available in Ref. 1. Ammonia-free water is used for analysis of ammonia. Ion-free water is suitable for the same purpose as well as determining trace metals and low concentrations of most ions. It is not suitable for some organic analysis because organic contaminants are added to the water during its treatment. Carbon dioxide-free water is used in carbon dioxide determinations. It is not advisable to store carbon dioxide-free water for long periods.

Compressed Air

- Analytical laboratories usually require high quality compressed air. Oil, water and dirt are undesirable contaminants and should be removed by filtering the flow from the compressors.

- High quality compressed air is available in cylinders.

Electric Services

- An adequate electric system including both 115- and 230-volt sources in sufficient capacity for the type of work that must be done, is indispensable to the analytical laboratory. If necessary for the proper functioning of electronic equipment, a constant voltage source must be available. For example, spectrophotometers, flame photometers, atomic absorption equipment, gas chromographs, etc. all need constant voltage to prevent drift.

- The lighting in a laboratory must be sufficient to accurately read glassware graduations, balance verniers, titration endpoints, etc.

Most Commonly Used Instruments in Water and Wastewater Analysis

ANALYTICAL BALANCE
pH METERS
CONDUCTIVITY METERS
TURBIDIMETERS
SPECTROPHOTOMETERS
ATOMIC SPECTROSCOPIC INSTRUMENTS
ORGANIC CARBON ANALYZER
ION-SELECTIVE ELECTRODES
GAS-LIQUID CHROMATOGRAPHS
DISSOLVED OXYGEN PROBES/METERS

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